

10670409-isocyanate

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10670409-isocyanate

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FILE COVERS 1907 - 21 Jun 2004 VOL 140 ISS 26  
FILE LAST UPDATED: 20 Jun 2004 (20040620/ED)

This file contains CAS Registry Numbers for easy and accurate substance identification.

- => s diisocyanate → product  
L1 43031 DIISOCYANATE
- => s diamine → reactant  
L2 39888 DIAMINE
- => s 11 and 12  
L3 1871 L1 AND L2 ←
- => s phosgene ← reactant  
L4 12338 PHOSGENE
- => s 14 and 13  
L5 64 L4 AND L3 ←
- => s "hydrogen chloride"  
823591 "HYDROGEN"  
991591 "CHLORIDE"  
L6 26359 "HYDROGEN CHLORIDE"  
("HYDROGEN" (W) "CHLORIDE") ← reactant
- => s 16 and 15  
L7 5 L6 AND L5 ← reference  
← general Rxn name
- => s phosgenation  
L8 1238 PHOSGENATION
- => s 18 and 15  
L9 31 L8 AND L5 ←
- => s 19 or 15 or 17  
L10 64 L9 OR L5 OR L7 ← fatal with Rxn + ~~other~~ Product
- => s spray  
L11 110287 SPRAY in water or air
- => s 111 and 110  
L12 0 L11 AND L10 ← required in improvement
- => s "spray nozzles"  
110287 "SPRAY"  
24235 "NOZZLES"  
L13 1621 "SPRAY NOZZLES"  
("SPRAY" (W) "NOZZLES") ←

10670409-isocyanate

=> s l13 and l10  
L14 0 L13 AND L10

=> s l13 and 13  
L15 0 L13 AND L3

=> s bib abs 1-31 19  
MISSING OPERATOR 1-31 L9  
The search profile that was entered contains terms or  
nested terms that are not separated by a logical operator.

=> d bib abs 1-31 19

L9 ANSWER 1 OF 31 CAPLUS COPYRIGHT 2004 ACS on STN  
AN 2004:261001 CAPLUS  
DN 140:287841  
TI Process for the preparation of polyisocyanates of the diphenylmethane series by **phosgenation** of nonneutralized polyamines of the diphenylmethane series which are prepared by the reaction of aniline with aqueous formaldehyde in the presence of HCl  
IN Koch, Daniel; Pirkl, Hans-Georg; Hagen, Torsten; Wershofen, Stefan  
PA Bayer Materialscience A.-G., Germany  
SO Eur. Pat. Appl., 7 pp.  
CODEN: EPXXDW  
DT Patent

LA German  
FAN.CNT 1

PATE

— — —

PT EP 1

|     |  |    |          |
|-----|--|----|----------|
| R:  | AT, BE, CH, DE, DK, ES,<br>IE, SI, LT, LV, FI, RO, |    |          |
| DE  | 10245703   | A1 | 20040401 |
| US  | 2004092701   | A1 | 20040513 |
| JP  | 2004123746   | A2 | 20040422 |
| BRN | DE 2003 10245703                                   | A  | 20030201 |

PRAI DE 2002-10245703 A 20020930  
AB Polyisocyanates of the diphenylmethane series (e.g., MDI) are prepared by **phosgenation** of nonneutralized polyamines (e.g., the corresponding **diamine** of MDI) of the diphenylmethane series which are prepared by the reaction of aniline with aqueous formaldehyde in the presence of HCl, where distillation of the water from the formed **diamine** before **phosgenation** is a process step.

RE.CNT 2 THERE ARE 2 CITED REFERENCES AVAILABLE FOR THIS RECORD  
ALL CITATIONS AVAILABLE IN THE RE FORMAT

L9 ANSWER 2 OF 31 CAPLUS COPYRIGHT 2004 ACS on STN

AN 2004:159009 CAPLUS

DN 140:199902

TI Process for the manufacture of aromatic diisocyanates via the gas-phase phosgenation of aromatic diamines under moderate pressures

IN Woelfert, Andreas; Mueller, Christian; Stroefer, Eckhard; Weber, Markus;  
Pfeffinger, Joachim; Knoesche, Carsten

PA BASF A.-G., Germany

SO Ger. Offen., 7 pp.

CODEN: GWXXBX

DT Patent

## LA German

FAN, CNT 1

PATED

- - - -

PT DE 1

WO 2004026813 A1 20040401

DE 2002-10238995 20020820  
WO 2003-EP8108 20030724

10670409-isocyanate

W: AE, AG, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, BZ, CA, CH, CN,  
CO, CR, CU, CZ, DE, DK, DM, DZ, EC, EE, ES, FI, GB, GD, GE, GH,  
GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR,  
LS, LT, LU, LV, MA, MD, MG, MK, MN, MW, MX, MZ, NI, NO, NZ, OM,  
PG, PH, PL, PT, RO, RU, SC, SD, SE, SG, SK, SL, SY, TJ, TM, TN,  
TR, TT, TZ, UA, UG, US, UZ, VC, VN, YU, ZA, ZM, ZW, AM, AZ, BY,  
KG, KZ, MD, RU  
RW: GH, GM, KE, LS, MW, MZ, SD, SL, SZ, TZ, UG, ZM, ZW, AT, BE, BG,  
CH, CY, CZ, DE, DK, EE, ES, FI, FR, GB, GR, HU, IE, IT, LU, MC,  
NL, PT, RO, SE, SI, SK, TR, BF, BJ, CF, CG, CI, CM, GA, GN, GQ,  
GW, ML, MR, NE, SN, TD, TG

PRAI DE 2002-10238995 A 20020820

AB Aromatic diisocyanates (e.g., TDI) are prepared in high yield and selectivity the gas-phase reaction of **phosgene** with aromatic diamines (e.g., toluenediamine) under moderate pressures of >3 bars and <20 bars. A process flow diagram is presented.

L9 ANSWER 3 OF 31 CAPLUS COPYRIGHT 2004 ACS on STN

AN 2003:950981 CAPLUS

DN 140:5423

TI **Phosgenation** method and reactors for producing isocyanates from **phosgene** and primary amines

IN Woelfert, Andreas; Mueller, Christian; Stroefer, Eckhard; Brodhagen, Andreas

PA Basf Aktiengesellschaft, Germany

SO PCT Int. Appl., 18 pp.

CODEN: PIXXD2

DT Patent

LA German

FAN.CNT 1

|    | PATENT NO.  | KIND  | DATE     | APPLICATION NO. | DATE     |
|----|---|---|----------|-----------------|----------|
| PI | WO 2003099770   | A1  | 20031204 | WO 2003-EP5232  | 20030519 |
|    | W: AE, AG, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, BZ, CA, CH, CN,<br>CO, CR, CU, CZ, DE, DK, DM, DZ, EC, EE, ES, FI, GB, GD, GE, GH,<br>GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR,<br>LS, LT, LU, LV, MA, MD, MG, MK, MN, MW, MX, MZ, NI, NO, NZ, OM,<br>PH, PL, PT, RO, RU, SC, SD, SE, SG, SK, SL, TJ, TM, TN, TR, TT,<br>TZ, UA, UG, US, UZ, VC, VN, YU, ZA, ZM, ZW, AM, AZ, BY, KG, KZ,<br>MD, RU, TJ, TM | RW: GH, GM, KE, LS, MW, MZ, SD, SL, SZ, TZ, UG, ZM, ZW, AT, BE, BG,<br>CH, CY, CZ, DE, DK, EE, ES, FI, FR, GB, GR, HU, IE, IT, LU, MC,<br>NL, PT, RO, SE, SI, SK, TR, BF, BJ, CF, CG, CI, CM, GA, GN, GQ,<br>GW, ML, MR, NE, SN, TD, TG |          |                 |          |

DE 10222968 A1 20031204 DE 2002-10222968 20020523

PRAI DE 2002-10222968 A 20020523

AB A continuous method for producing isocyanates (e.g., TDI) by reacting primary amines (e.g., toluenediamines) with **phosgene** is described in which the reaction is carried out in a cascade of at least two tubular reactors and, after each reactor, the gas phase resulting during the reaction is separated in a phase separator, and only the liquid phase

is fed to the next reactor or to the product purification In addition, the reaction volume of the first tubular reactor is equal to only a fraction of the total reaction volume

RE.CNT 5 THERE ARE 5 CITED REFERENCES AVAILABLE FOR THIS RECORD  
ALL CITATIONS AVAILABLE IN THE RE FORMAT

L9 ANSWER 4 OF 31 CAPLUS COPYRIGHT 2004 ACS on STN

AN 2003:910219 CAPLUS

DN 139:381885

TI **Phosgenation** process and reactor for the gas-phase preparation

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of isocyanates from their corresponding amines  
IN Jenne, Marc; Herold, Heiko; Friedrich, Martin; Stutz, Herbert  
PA Bayer Aktiengesellschaft, Germany  
SO Eur. Pat. Appl., 6 pp.  
CODEN: EPXXDW

DT Patent  
LA German

FAN.CNT 1

|      | PATENT NO.  | KIND | DATE     | APPLICATION NO.  | DATE     |
|------|---|------|----------|--|----------|
| PI   | EP 1362847  | A2   | 20031119 | EP 2003-10096  | 20030505 |
|      | EP 1362847  | A3   | 20040204 | R: AT, BE, CH, DE, DK, ES, FR, GB, GR, IT, LI, LU, NL, SE, MC, PT,<br>IE, SI, LT, LV, FI, RO, MK, CY, AL, TR, BG, CZ, EE, HU, SK |          |
|      | DE 10222023   | A1   | 20031127 | DE 2002-10222023   | 20020517 |
|      | US 2003216597   | A1   | 20031120 | US 2003-437509   | 20030514 |
|      | CN 1458150  | A    | 20031126 | CN 2003-136025   | 20030516 |
|      | JP 2004067669   | A2   | 20040304 | JP 2003-138482   | 20030516 |
| PRAI | DE 2002-10222023  | A    | 20020517 |  |          |
| OS   | MARPAT 139:381885   |      |          |  |          |
| AB   | A tubular <b>phosgenation</b> reactor, which exhibits reduced temperature fluctuations and thus has reduced polymeric buildup, is described as is a process for the gas-phase <b>phosgenation</b> of diamines (e.g., hexamethylenediamine) or triamines into their corresponding isocyanates (e.g., hexamethylenediisocyanate). |      |          |  |          |

L9 ANSWER 5 OF 31 CAPLUS COPYRIGHT 2004 ACS on STN

AN 2003:892453 CAPLUS

DN 139:382379

TI Manufacture of tetralin isocyanates as monomers for polyurethanes

IN Adkins, Rick L.; Parsons, Harold R.

PA Bayer Polymers, LLC, USA

SO U.S. Pat. Appl. Publ., 3 pp.

CODEN: USXXCO

DT Patent

LA English

FAN.CNT 1

|    | PATENT NO.    | KIND | DATE     | APPLICATION NO.   | DATE     |
|----|---------------|------|----------|---|----------|
| PI | US 2003212160 | A1   | 20031113 | US 2002-142289  | 20020509 |
|    | US 6750367    | B2   | 20040615 |   |          |
|    | WO 2003095419 | A1   | 20031120 | WO 2003-US14011   | 20030505 |
|    |               |      |          | W: AE, AG, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, BZ, CA, CH, CN,<br>CO, CR, CU, CZ, DE, DK, DM, DZ, EC, EE, ES, FI, GB, GD, GE, GH,<br>GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR,<br>LS, LT, LU, LV, MA, MD, MG, MK, MN, MW, MX, MZ, NI, NO, NZ, OM,<br>PH, PL, PT, RO, RU, SC, SD, SE, SG, SK, SL, TJ, TM, TN, TR, TT,<br>TZ, UA, UG, UZ, VC, VN, YU, ZA, ZM, ZW, AM, AZ, BY, KG, KZ, MD,<br>RU, TJ, TM |          |
|    |               |      |          | RW: GH, GM, KE, LS, MW, MZ, SD, SL, SZ, TZ, UG, ZM, ZW, AT, BE, BG,<br>CH, CY, CZ, DE, DK, EE, ES, FI, FR, GB, GR, HU, IE, IT, LU, MC,<br>NL, PT, RO, SE, SI, SK, TR, BF, BJ, CF, CG, CI, CM, GA, GN, GQ,<br>GW, ML, MR, NE, SN, TD, TG   |          |

PRAI US 2002-142289 A 20020509

AB Novel aromatic isocyanates, useful as isocyanate components in the production of

flexible polyurethane foam, are obtained by **phosgenation** of diaminotetralin in 50/50 diglyme/1,2-dichlorobenzene mixture. The latter **diamine** was prepared by nitration of tetralin and reduction of dinitrotetralin. Thus, polyurethane foam manufactured from tetralin **diisocyanate** 100, glycerol/propylene glycol-based polyol 100, H<sub>2</sub>O 4.40, L620 (silicone surfactant) 0.8 and Polycat 70 catalyst 1.0 part had

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d. 1.31 lb/ft<sup>3</sup>, tensile strength 17.3 lb/in<sup>2</sup>, elongation 208% and tear strength 2.13 lb/in, vs. 1.30, 17.7, 168 and 1.72, resp., for polyurethane foam manufactured with 100 parts TDI instead of tetralin **diisocyanate**

L9 ANSWER 6 OF 31 CAPLUS COPYRIGHT 2004 ACS on STN

AN 2003:434519 CAPLUS

DN 139:22615

TI Production of isocyanates in the gaseous phase

IN Woelfert, Andreas; Mueller, Christian; Stroefer, Eckhard; Pfeffinger, Joachim; Weber, Markus; Knoesche, Carsten

PA BASF Aktiengesellschaft, Germany

SO PCT Int. Appl., 21 pp.

CODEN: PIXXD2

DT Patent

LA German

FAN.CNT 1

| PATENT NO.  | KIND   | DATE     | APPLICATION NO. | DATE     |
|---|--|----------|-----------------|----------|
| PI WO 2003045900  | A1   | 20030605 | WO 2002-EP12930 | 20021119 |
| W: AE, AG, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, BZ, CA, CH, CN, CO, CR, CU, CZ, DE, DK, DM, DZ, EC, EE, ES, FI, GB, GD, GE, GH, GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MA, MD, MG, MK, MN, MW, MX, MZ, NO, NZ, OM, PH, PL, PT, RO, RU, SC, SD, SE, SG, SI, SK, SL, TJ, TM, TN, TR, TT, TZ, UA, UG, US, UZ, VC, VN, YU, ZA, ZM, ZW, AM, AZ, BY, KG, KZ, MD, RU, TJ, TM | RW: GH, GM, KE, LS, MW, MZ, SD, SL, SZ, TZ, UG, ZM, ZW, AT, BE, BG, CH, CY, CZ, DE, DK, EE, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE, SK, TR, BF, BJ, CF, CG, CI, CM, GA, GN, GQ, GW, ML, MR, NE, SN, TD, TG |          |                 |          |

DE 10158160 A1 20030612 DE 2001-10158160 20011128

PRAI DE 2001-10158160 A 20011128

AB The invention relates to a method for producing diisocyanates by reacting primary diamines with **phosgene** in the gaseous phase. Said method is characterized in that the reaction of **diamine** and **phosgene** occurs in a reaction channel, the internal dimensions of which have a width/height ratio of at least 2/1. With these dimensions the reaction chamber is useful for a longer period of time before it is necessary to clean the chamber of solid precipitate

RE.CNT 2 THERE ARE 2 CITED REFERENCES AVAILABLE FOR THIS RECORD  
ALL CITATIONS AVAILABLE IN THE RE FORMAT

L9 ANSWER 7 OF 31 CAPLUS COPYRIGHT 2004 ACS on STN

AN 2003:40165 CAPLUS

DN 138:90237

TI Process and apparatus for the preparation of (cyclo)aliphatic di- and triisocyanates by the gas-phase **phosgenation** of the corresponding diamines and triamines

IN Leimkuehler, Hans-Joachim; Stutz, Herbert; Schmidt, Helmut

PA Bayer AG, Germany

SO Eur. Pat. Appl., 7 pp.

CODEN: EPXXDW

DT Patent

LA German

FAN.CNT 1

| PATENT NO.  | KIND        | DATE     | APPLICATION NO. | DATE                      |
|---|-------------|----------|-----------------|---------------------------|
| PI EP 1275640   | A1          | 20030115 | EP 2002-14039   | 20020628                  |
| R: AT, BE, CH, DE, DK, ES, FR, GB, GR, IT, LI, LU, NL, SE, MC, PT, IE, SI, LT, LV, FI, RO, MK, CY, AL, TR | DE 10133728 | A1       | 20030123        | DE 2001-10133728 20010711 |

10670409-isocyanate

|               |    |          |                |          |
|---------------|----|----------|----------------|----------|
| US 2003069441 | A1 | 20030410 | US 2002-190262 | 20020703 |
| US 6706913    | B2 | 20040316 |                |          |
| JP 2003096043 | A2 | 20030403 | JP 2002-195908 | 20020704 |
| CN 1396153    | A  | 20030212 | CN 2002-140950 | 20020711 |

PRAI DE 2001-10133728 A 20010711

OS MARPAT 138:90237

AB (cyclo)aliphatic di- (e.g., 1,6-diisocyanatohexane) and triisocyanates are prepared in high yield and selectivity by the gas-phase **phosgenation** of the corresponding diamines (e.g., 1,6-diaminohexane) and triamines at 200-600° where the **phosgene** is injected into the vaporized amine stream at a point in the reactor where the reactor internal diameter is narrowed and then re-expands past the point of the **phosgenation** reaction. An apparatus diagram is presented.

RE.CNT 1 THERE ARE 1 CITED REFERENCES AVAILABLE FOR THIS RECORD  
ALL CITATIONS AVAILABLE IN THE RE FORMAT

L9 ANSWER 8 OF 31 CAPLUS COPYRIGHT 2004 ACS on STN

AN 2003:40164 CAPLUS

DN 138:90236

TI Process and apparatus for the preparation of (cyclo)aliphatic di- and triisocyanates by the gas-phase **phosgenation** of the corresponding diamines and triamines

IN Leimkuehler, Hans-Joachim; Stutz, Herbert; Leuckel, Wolfgang

PA Bayer Aktiengesellschaft, Germany

SO Eur. Pat. Appl., 9 pp.

CODEN: EPXXDW

DT Patent

LA German

FAN.CNT 1

|    | PATENT NO.   | KIND | DATE     | APPLICATION NO.  | DATE     |
|----|--|------|----------|------------------|----------|
| PI | EP 1275639   | A1   | 20030115 | EP 2002-14038    | 20020628 |
|    | R: AT, BE, CH, DE, DK, ES, FR, GB, GR, IT, LI, LU, NL, SE, MC, PT,<br>IE, SI, LT, LV, FI, RO, MK, CY, AL, TR |      |          |                  |          |
|    | DE 10133729  | A1   | 20030123 | DE 2001-10133729 | 20010711 |
|    | US 2003013909  | A1   | 20030116 | US 2002-190265   | 20020703 |
|    | JP 2003104948  | A2   | 20030409 | JP 2002-195902   | 20020704 |
|    | CN 1396152   | A    | 20030212 | CN 2002-140947   | 20020711 |

PRAI DE 2001-10133729 A 20010711

OS MARPAT 138:90236

AB (cyclo)aliphatic di- (e.g., 1,6-diisocyanatohexane) and triisocyanates are prepared in high yield and selectivity by the gas-phase **phosgenation** of the corresponding diamines (e.g., 1,6-diaminohexane) and triamines at 200-600° where the **phosgene** is injected into the gaseous amine stream at a point in the reactor where the reactor internal diameter is narrowed and then re-expands past the point of the **phosgenation** reaction. An apparatus diagram is presented.

RE.CNT 6 THERE ARE 6 CITED REFERENCES AVAILABLE FOR THIS RECORD  
ALL CITATIONS AVAILABLE IN THE RE FORMAT

L9 ANSWER 9 OF 31 CAPLUS COPYRIGHT 2004 ACS on STN

AN 2001:488630 CAPLUS

DN 135:61735

TI Method for making aliphatic diisocyanates which uses supercritical extraction of the process waste stream

IN Sommer, Alexa B.; Wittig, Mary Ann; Hortelano, Edwin R.; Yeske, Philip E.; Ciebien, Jane F.

PA Bayer Corporation, USA

SO Eur. Pat. Appl., 14 pp.

CODEN: EPXXDW

DT Patent

LA English

10670409-isocyanate

FAN.CNT 1

|    | PATENT NO. | KIND | DATE     | APPLICATION NO.  | DATE     |
|----|------------|------|----------|--|----------|
| PI | EP 1113003 | A1   | 20010704 | EP 2000-127231   | 20001215 |
|    | EP 1113003 | B1   | 20030820 | R: AT, BE, CH, DE, DK, ES, FR, GB, GR, IT, LI, LU, NL, SE, MC, PT,<br>IE, SI, LT, LV, FI, RO |          |
|    | US 6307096 | B1   | 20011023 | US 1999-473097   | 19991228 |
|    | AT 247636  | E    | 20030915 | AT 2000-127231   | 20001215 |

PRAI US 1999-473097 A 19991228

AB A method for making an aliphatic **diisocyanate** (e.g., hexamethylene **diisocyanate**) is described comprising: (a) phosgenating an aliphatic **diamine** (e.g., 1,6-diaminohexane) in the presence of an inert solvent or gas to form a crude reaction mixture; (b) distilling the crude reaction mixture to form an aliphatic **diisocyanate** production stream and an aliphatic **diisocyanate** waste stream; (c) introducing the aliphatic **diisocyanate** waste stream to a chamber and placing the waste stream under supercrit. fluid conditions sufficient to dissolve an appreciable amount of the aliphatic **diisocyanate** component in the supercrit. fluid (e.g., carbon dioxide); (d) separating the dissolved aliphatic **diisocyanate** component from the waste stream, where the remaining waste stream is a supercritically-purged aliphatic **diisocyanate** waste stream; and (e) lowering the pressure sufficiently to precipitate the aliphatic **diisocyanate** component.

RE.CNT 4 THERE ARE 4 CITED REFERENCES AVAILABLE FOR THIS RECORD  
ALL CITATIONS AVAILABLE IN THE RE FORMAT

L9 ANSWER 10 OF 31 CAPLUS COPYRIGHT 2004 ACS on STN  
AN 2001:165790 CAPLUS  
DN 134:207403  
TI Improved procedure for the production of mono- and oligoisocyanates by the **phosgenation** of primary amines in the presence of catalytic amounts of monoisocyanates  
IN Stamm, Armin; Kneuper, Heinz-josef; Thil, Lucien; Henkelmann, Jochem  
PA BASF AG, Germany  
SO Ger. Offen., 8 pp.  
CODEN: GWXXBX  
DT Patent  
LA German

FAN.CNT 1

|      | PATENT NO.   | KIND | DATE     | APPLICATION NO.  | DATE     |
|------|--|------|----------|--|----------|
| PI   | DE 19942299  | A1   | 20010308 | DE 1999-19942299   | 19990904 |
|      | WO 2001017951  | A1   | 20010315 | WO 2000-EP8221   | 20000823 |
|      | W: AE, AG, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, BZ, CA, CH, CN,<br>CR, CU, CZ, DE, DK, DM, DZ, EE, ES, FI, GB, GD, GE, GH, GM, HR,<br>HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT,<br>LU, LV, MA, MD, MG, MK, MN, MW, MX, MZ, NO, NZ, PL, PT, RO, RU,<br>SD, SE, SG, SI, SK, SL, TJ, TM, TR, TT, TZ, UA, UG, US, UZ, VN,<br>YU, ZA, ZW, AM, AZ, BY, KG, KZ, MD, RU, TJ, TM<br>RW: GH, GM, KE, LS, MW, MZ, SD, SL, SZ, TZ, UG, ZW, AT, BE, CH, CY,<br>DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE, BF, BJ,<br>CF, CG, CI, CM, GA, GN, GW, ML, MR, NE, SN, TD, TG |      |          |  |          |
|      | EP 1208082   | A1   | 20020529 | EP 2000-951530   | 20000823 |
|      | EP 1208082   | B1   | 20040407 | R: AT, BE, CH, DE, DK, ES, FR, GB, GR, IT, LI, LU, NL, SE, MC, PT,<br>IE, SI, LT, LV, FI, RO, MK, CY, AL |          |
|      | AT 263752  | E    | 20040415 | AT 2000-951530   | 20000823 |
|      | US 6683204   | B1   | 20040127 | US 2002-70393  | 20020304 |
| PRAI | DE 1999-19942299   | A    | 19990904 |  |          |
|      | WO 2000-EP8221   | W    | 20000823 |  |          |
| OS   | CASREACT 134:207403; MARPAT 134:207403   |      |          |  |          |

10670409-isocyanate

AB Aliphatic, cycloaliph., araliph. [e.g., R-(+)-phenylethyl isocyanate], or aromatic mono- and oligoisocyanates are prepared in high yield and selectivity by the **phosgenation** of the appropriate primary amines [e.g., R-(+)-phenylethylamine] at atmospheric pressure with **phosgene** in the presence of catalytic amounts of monoisocyanates (e.g., Bu isocyanate) in an inert solvent (e.g., chlorobenzene).

L9 ANSWER 11 OF 31 CAPLUS COPYRIGHT 2004 ACS on STN

AN 1998:629973 CAPLUS

DN 129:231150

TI Process for the preparation of diisocyanates of light color by the **phosgenation** of diamines treated with inorganic solids having Lewis and/or Bronsted acid centers

IN Kraus, Rupert; Reif, Martin; Bruchmann, Bernd; Tesch, Helmut

PA BASF A.-G., Germany

SO Eur. Pat. Appl., 5 pp.

CODEN: EPXXDW

DT Patent

LA German

FAN.CNT 1

|      | PATENT NO.  | KIND | DATE     | APPLICATION NO.  | DATE     |
|------|---|------|----------|------------------|----------|
| PI   | EP 866057   | A2   | 19980923 | EP 1998-103997   | 19980306 |
|      | EP 866057   | A3   | 20020814 |                  |          |
|      | R: AT, BE, CH, DE, DK, ES, FR, GB, GR, IT, LI, LU, NL, SE, MC, PT, IE, SI, LT, LV, FI, RO |      |          |                  |          |
|      | DE 19711447   | A1   | 19980924 | DE 1997-19711447 | 19970319 |
|      | CA 2229835  | AA   | 19980919 | CA 1998-2229835  | 19980318 |
|      | US 5872278  | A    | 19990216 | US 1998-40673    | 19980318 |
|      | CN 1197793  | A    | 19981104 | CN 1998-108727   | 19980319 |
|      | CN 1070475  | B    | 20010905 |                  |          |
|      | JP 10306068   | A2   | 19981117 | JP 1998-70194    | 19980319 |
| PRAI | DE 1997-19711447  | A    | 19970319 |                  |          |

AB Diisocyanates (e.g., MDI) having little discoloration, suitable as monomers for the manufacture of polyurethanes (no data), are prepared by the treatment of diamines [e.g., 1,1'-methylenebis(4-aminobenzene)] with inorg. solids which contain Lewis and/or Bronsted acid centers (e.g., mol. sieves) followed by **phosgenation** of the treated diamines.

L9 ANSWER 12 OF 31 CAPLUS COPYRIGHT 2004 ACS on STN

AN 1996:248012 CAPLUS

DN 124:261969

TI Continuous manufacture of aromatic diisocyanates

IN Bestiuc, Ioan; Caraculacu, Adrian; Idriceanu, Silvia; Buruiana, Tinca; Kelemen, Zoltan; Pogor, Constantin

PA Inst. de Chimie Macromoleculara "Petru Poni", Iasi, Rom.

SO Rom., 6 pp.

CODEN: RUXXA3

DT Patent

LA Romanian

FAN.CNT 1

|      | PATENT NO.     | KIND | DATE     | APPLICATION NO. | DATE     |
|------|----------------|------|----------|-----------------|----------|
| PI   | RO 104788      | B1   | 19940920 | RO 1989-143106  | 19891211 |
| PRAI | RO 1989-143106 |      | 19891211 |                 |          |

AB Aromatic diisocyanates are manufactured by intimately contacting a solution of 5-20% aromatic diamines in a solvent selected from hydrocarbons, chloride compds., esters, or ethers with COCl<sub>2</sub> at COCl<sub>2</sub>-diamine mol ratio >2.5

using a pump mounted on the shaft of a stirrer, with recirculation of a HCl-COCl<sub>2</sub> gas mixture from the upper part of the reactor through the reaction mixture. This method provides product with low concentration of

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hydrolyzable Cl and eliminates the requirement of 1st forming hydrochlorides of the diamines.

L9 ANSWER 13 OF 31 CAPLUS COPYRIGHT 2004 ACS on STN  
AN 1995:994743 CAPLUS  
DN 124:86379  
TI Process for the preparation of diisocyanates by the **phosgenation** of diamines  
IN Bischoff, Eric; Breidenbach, Peter; Dahmer, Juergen; Flink, Andreas;  
Molnar, Attila; Stutz, Herbert  
PA Bayer A.-G., Germany  
SO Eur. Pat. Appl., 6 pp.  
CODEN: EPXXDW

DT Patent

LA German

FAN.CNT 1

|      | PATENT NO.                    | KIND | DATE     | APPLICATION NO. | DATE     |
|------|-------------------------------|------|----------|-----------------|----------|
| PI   | EP 676392                     | A1   | 19951011 | EP 1995-104637  | 19950329 |
|      | EP 676392                     | B1   | 20010620 |                 |          |
|      | R: BE, DE, ES, FR, GB, IT, NL |      |          |                 |          |
|      | DE 4412327                    | A1   | 19951012 | DE 1994-4412327 | 19940411 |
|      | EP 1078918                    | A1   | 20010228 | EP 2000-202871  | 19950329 |
|      | EP 1078918                    | B1   | 20021211 |                 |          |
|      | R: BE, DE, ES, FR, GB, IT, NL |      |          |                 |          |
|      | ES 2159579                    | T3   | 20011016 | ES 1995-104637  | 19950329 |
|      | ES 2188472                    | T3   | 20030701 | ES 2000-202871  | 19950329 |
|      | US 5516935                    | A    | 19960514 | US 1995-413646  | 19950330 |
|      | CA 2146522                    | AA   | 19951012 | CA 1995-2146522 | 19950406 |
|      | JP 07278089                   | A2   | 19951024 | JP 1995-107109  | 19950407 |
| PRAI | DE 1994-4412327               | A    | 19940411 |                 |          |
|      | EP 1995-104637                | A3   | 19950329 |                 |          |

AB Diisocyanates (e.g., 1,3-pentane **diisocyanate**) are prepared by the gas-phase **phosgenation** of the corresponding diamines (e.g., 1,3-diaminopentane) in which: gaseous diamines, if necessary diluted with inert gas or inert-solvent vapors, and **phosgene**, are sep. heated to 200-600° and continuously brought together in a non-stirred cyclindrical reactor heated to 200-600° by maintaining a turbulent stream so they react; the gas mixture continuously leaving the reactor with the help of an inert liquid solvent, that is kept at a temperature above the decomposition temperature of the carbamic acid chloride corresponding to the **diamine**, is condensed to give a solution of the **diisocyanate** in this solvent; and the desired **diisocyanate** dissolved in the inert solvent is recovered by distillation

L9 ANSWER 14 OF 31 CAPLUS COPYRIGHT 2004 ACS on STN  
AN 1995:992543 CAPLUS  
DN 124:88109  
TI Preparation of aliphatic polyisocyanates from polyamines and **phosgene**  
PA Mitsui Toatsu Chemicals, Inc., Japan  
SO Ger. Offen., 11 pp.  
CODEN: GWXXBX

DT Patent

LA German

FAN.CNT 1

|    | PATENT NO.  | KIND | DATE     | APPLICATION NO.  | DATE     |
|----|-------------|------|----------|------------------|----------|
| PI | DE 19510259 | A1   | 19950928 | DE 1995-19510259 | 19950321 |
|    | DE 19510259 | C2   | 19970904 |                  |          |
|    | JP 07309827 | A2   | 19951128 | JP 1995-42956    | 19950302 |
|    | JP 3201921  | B2   | 20010827 |                  |          |

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|            |   |          |                |          |
|------------|---|----------|----------------|----------|
| US 5523467 | A | 19960604 | US 1995-401807 | 19950310 |
| CN 1125718 | A | 19960703 | CN 1995-104546 | 19950322 |
| CN 1062857 | B | 20010307 |                |          |

PRAI JP 1994-50082 A 19940322

AB In the conversion of an aliph polyamine to the polyisocyanate (e.g., m-xylylenediamine to m-xylylene **diisocyanate**) in an inert liquid medium, an inert gas is added to the reactor during the reaction to increase the yield of polyisocyanate and reduce the amount of **phosgene** required.

L9 ANSWER 15 OF 31 CAPLUS COPYRIGHT 2004 ACS on STN  
AN 1995:103343 CAPLUS

DN 122:188201

TI 1,3-Pentane **diisocyanate**

AU Sheridan, Robert E.

CS E. I. Du Pont and Co., USA

SO Research Disclosure (1994), 362, 306 (No. 36220)  
CODEN: RSDSBB; ISSN: 0374-4353

DT Journal; Patent

LA English

| PATENT NO. | KIND  | DATE  | APPLICATION NO. | DATE  |
|------------|-------|-------|-----------------|-------|
| -----      | ----- | ----- | -----           | ----- |

PI RD 362020 19940610

PRAI RD 1994-362020 19940610

AB Direct hot **phosgenation** of 1,3-diaminopentane at 125° in m-chlorobenzene resulted in 30% yield of 1,3-pentane **diisocyanate**. Subsequent distillation after addition of di-ortho-xylylethane resulted in

34%

recovery (10% based on starting **diamine**) of 1,3-pentane **diisocyanate** which was 82-85% pure. The product was characterized by IR and mass spectroscopy.

L9 ANSWER 16 OF 31 CAPLUS COPYRIGHT 2004 ACS on STN

AN 1994:701572 CAPLUS

DN 121:301572

TI Manufacture of diphenylmethane 4,4'-**diisocyanate**

IN Botezatu, Petru; Miron, Ada Margareta; Olanescu, Emil; Stoica, Dumitru; Teodorescu, Dan Cezar

PA Combinatul de Fibre Sintetice, Savinesti, Rom.

SO Rom., 3 pp.

CODEN: RUXXA3

DT Patent

LA Romanian

FAN.CNT 1

| PATENT NO. | KIND  | DATE  | APPLICATION NO. | DATE  |
|------------|-------|-------|-----------------|-------|
| -----      | ----- | ----- | -----           | ----- |

PI RO 103439 B1 19921123 RO 1989-138956 19890329

PRAI RO 1989-138956 19890329

AB Title compound, useful for the manufacture of polyurethanes (no data), is prepared

by **phosgenation** of the dihydrochloride of isomer mixts. containing 96-98% 4,4'-diaminodiphenylmethane 8 h in polyalkylbenzene at 140-160° and CO<sub>2</sub>Cl/**diamine** = 1-1.5/1.

L9 ANSWER 17 OF 31 CAPLUS COPYRIGHT 2004 ACS on STN

AN 1994:192549 CAPLUS

DN 120:192549

TI Preparation of aromatic diisocyanates by **phosgenation** in gas phase

IN Biskup, Klaus; Koenig, Christian; Waldau, Eckart

PA Bayer A.-G., Germany

SO Eur. Pat. Appl., 6 pp.

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CODEN: EPXXDW

DT Patent

LA German

FAN.CNT 1

|      | PATENT NO.            | KIND | DATE     | APPLICATION NO. | DATE     |
|------|-----------------------|------|----------|-----------------|----------|
| PI   | EP 570799             | A1   | 19931124 | EP 1993-107560  | 19930510 |
|      | EP 570799             | B1   | 19960619 |                 |          |
|      | R: BE, DE, FR, IT, NL |      |          |                 |          |
|      | DE 4217019            | A1   | 19931125 | DE 1992-4217019 | 19920522 |
|      | CA 2096501            | AA   | 19931123 | CA 1993-2096501 | 19930518 |
|      | CA 2096501            | C    | 20030729 |                 |          |
|      | BR 9302015            | A    | 19931130 | BR 1993-2015    | 19930520 |
|      | JP 06041046           | A2   | 19940215 | JP 1993-139885  | 19930520 |
|      | JP 3219903            | B2   | 20011015 |                 |          |
| PRAI | DE 1992-4217019       | A    | 19920522 |                 |          |
| OS   | MARPAT 120:192549     |      |          |                 |          |

AB Continuous **phosgenation** of arom diamines, e.g., a mixture of 2,4- and 2,6-diaminotoluenes or bis(4-aminophenyl)methane, in the gas phase with residence time 0.5-5 s gives high yields (e.g., >99%) of diisocyanates without deposition of solid material in the reactor.

L9 ANSWER 18 OF 31 CAPLUS COPYRIGHT 2004 ACS on STN

AN 1991:448872 CAPLUS

DN 115:48872

TI Preparation of aliphatic isocyanates by reaction of aliphatic amines with **phosgene** in esters as solvents

IN Nagata, Teruyuki; Wada, Masaru; Mizuta, Hideki

PA Mitsui Toatsu Chemicals, Inc., Japan

SO Eur. Pat. Appl., 8 pp.

CODEN: EPXXDW

DT Patent

LA English

FAN.CNT 1

|      | PATENT NO.                | KIND | DATE     | APPLICATION NO. | DATE     |
|------|---------------------------|------|----------|-----------------|----------|
| PI   | EP 424836                 | A1   | 19910502 | EP 1990-120195  | 19901022 |
|      | EP 424836                 | B1   | 19950315 |                 |          |
|      | R: BE, DE, FR, GB, IT, NL |      |          |                 |          |
|      | JP 03204851               | A2   | 19910906 | JP 1990-273283  | 19901015 |
|      | JP 2986888                | B2   | 19991206 |                 |          |
|      | CA 2028162                | AA   | 19910424 | CA 1990-2028162 | 19901022 |
|      | US 5136086                | A    | 19920804 | US 1992-819657  | 19920113 |
| PRAI | JP 1989-273811            |      | 19891023 |                 |          |
|      | JP 1989-275047            |      | 19891024 |                 |          |
|      | US 1990-598113            |      | 19901016 |                 |          |

OS CASREACT 115:48872

AB A process for the preparation of aliphatic polyisocyanates comprises the treatment

of an aliphatic polyamine or a hydrochloride thereof with COCl<sub>2</sub> in the presence of an ester as a solvent. A flask was charged with hexamethylenediamine (46.5 g) and MeCO<sub>2</sub>(CH<sub>2</sub>)<sub>5</sub>Me as a solvent (613.5 g) and 35 g HCl were blown over the mixture which was then heated to 150° and then COCl<sub>2</sub> was blown over the mix. at 29.4 g/h for 15 h; the yield of hexamethylene **diisocyanate** was 90%. The use of other solvents, e.g., mesitylene, gave a lower yield of hexamethylene **diisocyanate** and a higher yield of 6-chlorohexane **diisocyanate**.

L9 ANSWER 19 OF 31 CAPLUS COPYRIGHT 2004 ACS on STN

AN 1989:231172 CAPLUS

DN 110:231172

TI Procedure for the production of (cyclo)aliphatic diisocyanates

10670409-isocyanate

IN Frosch, Hans Georg; Grave, Heinrich; Stutz, Herbert; Waldau, Eckart;  
Fuhrmann, Peter

PA Bayer A.-G., Fed. Rep. Ger.

SO Ger. Offen., 4 pp.

CODEN: GWXXBX

DT Patent

LA German

FAN.CNT 1

|    | PATENT NO.                    | KIND | DATE     | APPLICATION NO. | DATE     |
|----|-------------------------------|------|----------|-----------------|----------|
| PI | DE 3714439                    | A1   | 19881110 | DE 1987-3714439 | 19870430 |
|    | EP 289840                     | A1   | 19881109 | EP 1988-106111  | 19880416 |
|    | EP 289840                     | B1   | 19901017 |                 |          |
|    | R: BE, DE, ES, FR, GB, IT, NL |      |          |                 |          |
|    | US 4847408                    | A    | 19890711 | US 1988-185721  | 19880425 |
|    | CA 1305165                    | A1   | 19920714 | CA 1988-565025  | 19880425 |
|    | JP 63280050                   | A2   | 19881117 | JP 1988-104461  | 19880428 |
|    | JP 08025984                   | B4   | 19960313 |                 |          |

PRAI DE 1987-3714439 19870430

OS CASREACT 110:231172; MARPAT 110:231172

AB A procedure for the preparation of OCNRNCO [R = C1-15(cyclo)aliphatic hydrocarbon

moiety] by **phosgenation** of the corresponding H<sub>2</sub>NRNH<sub>2</sub> in the gas phase was characterized in that one: a) brings the gaseous **diamine**, optionally diluted with an inert gas or the vapors of an inert solvent, and COCl<sub>2</sub>, sep. heated to 200-600°, into reaction with each other in a cylindrical chamber at 200-600° without moving parts with the maintenance of a turbulent streaming into the reactor chamber; b) leads the gas mixture which continuously leaves the reaction chamber through an inert solvent which is kept at a temperature above the decomposition temperature of the carbamoyl chloride corresponding to the **diamine**; and c) subjects the **diisocyanate** dissolved in the inert solvent to a distillative work-up. In this manner, COCl<sub>2</sub> and H<sub>2</sub>N(CH<sub>2</sub>)<sub>6</sub>NH<sub>2</sub> reacted at 400° to give 98.0% OCN(CH<sub>2</sub>)<sub>6</sub>NCO.

L9 ANSWER 20 OF 31 CAPLUS COPYRIGHT 2004 ACS on STN

AN 1988:591412 CAPLUS

DN 109:191412

TI Preparation of aromatic diisocyanates and their mixtures for use in polyurethanes

IN Koenig, Klaus; Heitkaemper, Peter

PA Bayer A.-G., Fed. Rep. Ger.

SO Ger. Offen., 10 pp.

CODEN: GWXXBX

DT Patent

LA German

FAN.CNT 1

|    | PATENT NO.                        | KIND | DATE     | APPLICATION NO. | DATE     |
|----|-----------------------------------|------|----------|-----------------|----------|
| PI | DE 3641702                        | A1   | 19880616 | DE 1986-3641702 | 19861206 |
|    | EP 270906                         | A1   | 19880615 | EP 1987-117283  | 19871124 |
|    | EP 270906                         | B1   | 19900221 |                 |          |
|    | R: BE, DE, ES, FR, GB, IT, NL, SE |      |          |                 |          |
|    | CA 1289146                        | A1   | 19910917 | CA 1987-553105  | 19871130 |
|    | US 4888125                        | A    | 19891219 | US 1987-128295  | 19871203 |
|    | JP 63159360                       | A2   | 19880702 | JP 1987-306093  | 19871204 |

PRAI DE 1986-3641702 19861206

AB Mixts. of the diisocyanates p-OCNC<sub>6</sub>H<sub>4</sub>O(CH<sub>2</sub>CH<sub>2</sub>)<sub>n</sub>OC<sub>6</sub>H<sub>3</sub>(Me)NCO-2,1 30-100, p-OCNC<sub>6</sub>H<sub>4</sub>O(CH<sub>2</sub>CH<sub>2</sub>)<sub>n</sub>OC<sub>6</sub>H<sub>4</sub>NCO-p 0-50, and 2,1-OCN(Me)C<sub>6</sub>H<sub>3</sub>O(CH<sub>2</sub>CH<sub>2</sub>)<sub>n</sub>OC<sub>6</sub>H<sub>3</sub>(Me)NCO-2,1 0-50% (n = 1-3), useful in the manufacture of high-quality polyurethanes, are prepared by phosgenating the corresponding diamines.

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Adding 1348 g 2-(4-nitrophenoxy)ethyl p-toluenesulfonate in portions to 612 g 3-methyl-4-nitrophenol and 760 g 30% NaOMe in 6 L ethylene glycol at 80° and stirring 6 h at 100° gave 1029 g 1-(3-methyl-4-nitrophenoxy)-2-(4-nitrophenoxy)ethane, hydrogenation of which in DMF over Raney Ni at 75-90°/40-50 bar gave 92% diamine, phosgenation of which in PhCl gave 96% diisocyanate (m.p. 86-87°) containing 70 ppm hydrolyzable Cl.

L9 ANSWER 21 OF 31 CAPLUS COPYRIGHT 2004 ACS on STN  
AN 1988:474090 CAPLUS  
DN 109:74090  
TI Preparation of a diisocyanate for use in plastics  
IN Schmidt, Manfred; Koenig, Klaus; Heitkaemper, Peter; Pedain, Josef  
PA Bayer A.-G., Fed. Rep. Ger.  
SO Ger. Offen., 6 pp.  
CODEN: GWXXBX  
DT Patent  
LA German  
FAN.CNT 1

|      | PATENT NO.   | KIND | DATE     | APPLICATION NO. | DATE     |
|------|--|------|----------|-----------------|----------|
| PI   | DE 3633712   | A1   | 19880414 | DE 1986-3633712 | 19861003 |
|      | CA 1296350   | A1   | 19920225 | CA 1987-547055  | 19870916 |
|      | EP 265668  | A1   | 19880504 | EP 1987-113871  | 19870923 |
|      | EP 265668  | B1   | 19910109 |                 |          |
|      | R: BE, DE, FR, GB, IT, NL  |      |          |                 |          |
|      | JP 63091355  | A2   | 19880422 | JP 1987-245961  | 19871001 |
|      | US 5130466   | A    | 19920714 | US 1990-463713  | 19900111 |
| PRAI | DE 1986-3633712  |      | 19861003 |                 |          |
|      | US 1987-103081   |      | 19870930 |                 |          |
| AB   | The diisocyanate OCN(CH <sub>2</sub> ) <sub>5</sub> CO <sub>2</sub> CH <sub>2</sub> CMe <sub>2</sub> CH <sub>2</sub> NCO (I) is prepared by phosgenation of the corresponding diamine-2HCl (II). Adding 930 g H <sub>2</sub> N(CH <sub>2</sub> ) <sub>5</sub> COCl-HCl in 3.3 L CH <sub>2</sub> Cl <sub>2</sub> over 2 h to a HCl-saturated solution of 535 g H <sub>2</sub> NCH <sub>2</sub> C(Me) <sub>2</sub> CH <sub>2</sub> OH in 5 L CH <sub>2</sub> Cl <sub>2</sub> at 20° and heating at 40° gave 1420 g II. Passing COCl <sub>2</sub> at 50 g/h into a refluxing mixture of 289 g II and 3.5 L PhCl for 8 h gave 252 g I. |      |          |                 |          |

L9 ANSWER 22 OF 31 CAPLUS COPYRIGHT 2004 ACS on STN  
AN 1988:455421 CAPLUS  
DN 109:55421  
TI Preparation of special diisocyanates for use in polyurethanes  
IN Sanders, Josef  
PA Bayer A.-G., Fed. Rep. Ger.  
SO Ger. Offen., 11 pp.  
CODEN: GWXXBX  
DT Patent  
LA German  
FAN.CNT 1

|      | PATENT NO.                           | KIND | DATE     | APPLICATION NO. | DATE     |
|------|--------------------------------------|------|----------|-----------------|----------|
| PI   | DE 3628316                           | A1   | 19880225 | DE 1986-3628316 | 19860821 |
|      | EP 257420                            | A1   | 19880302 | EP 1987-111535  | 19870810 |
|      | EP 257420                            | B1   | 19900516 |                 |          |
|      | R: BE, DE, ES, FR, GB, IT, NL, SE    |      |          |                 |          |
|      | ES 2036198                           | T3   | 19930516 | ES 1987-111535  | 19870810 |
|      | US 4820866                           | A    | 19890411 | US 1987-84547   | 19870812 |
|      | CA 1292006                           | A1   | 19911112 | CA 1987-544617  | 19870814 |
|      | JP 63057564                          | A2   | 19880312 | JP 1987-205349  | 19870820 |
|      | US 4914238                           | A    | 19900403 | US 1989-320678  | 19890308 |
| PRAI | DE 1986-3628316                      |      | 19860821 |                 |          |
|      | US 1987-84547                        |      | 19870812 |                 |          |
| OS   | CASREACT 109:55421; MARPAT 109:55421 |      |          |                 |          |

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AB The title diisocyanates Z[OC<sub>6</sub>H<sub>3</sub>(R)NCO]<sub>2</sub> [R = H, Me; Z = (oxa)alkylene] are prepared by the reaction of Z(OH)<sub>2</sub> or O<sub>2</sub>N(R)C<sub>6</sub>H<sub>3</sub>OZOH with O<sub>2</sub>N(R)C<sub>6</sub>H<sub>3</sub>Cl, reduction of NO<sub>2</sub> groups, and **phosgenation**. Condensing 1 mol neopentyl glycol with 2.2 mol p-O<sub>2</sub>NC<sub>6</sub>H<sub>4</sub>Cl in DMSO containing powdered NaOH at 40-50° gave 92.8% 2,2-dimethyl-1,3-bis(4-nitrophenoxy)propane, hydrogenation of which in DMF over Raney Ni at 60°/50 bar gave 91% **diamine**. **Phosgenation** of the **diamine** in PhCl at -10° to 0° and refluxing gave 71.3% **diisocyanate** containing 0.007% hydrolyzable Cl.

L9 ANSWER 23 OF 31 CAPLUS COPYRIGHT 2004 ACS on STN

AN 1988:455417 CAPLUS

DN 109:55417

TI Aliphatic diisocyanates for preparation of polyurethanes

PA Bayer A.-G., Fed. Rep. Ger.

SO Jpn. Kokai Tokkyo Koho, 8 pp.

CODEN: JKXXAF

DT Patent

LA Japanese

FAN.CNT 2

|    | PATENT NO.  | KIND | DATE     | APPLICATION NO. | DATE     |
|----|-------------|------|----------|-----------------|----------|
| PI | JP 62221661 | A2   | 19870929 | JP 1987-54317   | 19870311 |
|    | DE 3608354  | A1   | 19870917 | DE 1986-3608354 | 19860313 |
|    | DE 3620821  | A1   | 19871223 | DE 1986-3620821 | 19860621 |
|    | DE 3620821  | C2   | 19940908 |                 |          |

PRAI DE 1986-3608354 19860313  
DE 1986-3620821 19860621

OS CASREACT 109:55417

AB Diisocyanates OCNCR<sub>1</sub>R<sub>2</sub>ZCH<sub>2</sub>NCO (R<sub>1</sub>, R<sub>2</sub> = C<sub>1-4</sub> alkyl; Z = C<sub>2-9</sub> aliphatic hydrocarbylene) are prepared. Thus, a solution of COCl<sub>2</sub> in PhCl was mixed with a solution of H<sub>2</sub>N(CH<sub>2</sub>)<sub>3</sub>CMe<sub>2</sub>NH<sub>2</sub> at 10-20° for 1 h under vigorous stirring, heated to 127° during 3 h with addition of COCl<sub>2</sub>, and refluxed 1 h at 127° to give 74% OCN(CH<sub>2</sub>)<sub>3</sub>CMe<sub>2</sub>NCO.

L9 ANSWER 24 OF 31 CAPLUS COPYRIGHT 2004 ACS on STN

AN 1987:197297 CAPLUS

Correction of: 1986:34839

DN 106:197297

Correction of: 104:34839

TI Low-melting diphenylethane **diisocyanate** for polyurethanes

IN Kervennal, Jacques; Mathais, Henri

PA Atochem S. A., Fr.

SO Eur. Pat. Appl., 12 pp.

CODEN: EPXXDW

DT Patent

LA French

FAN.CNT 1

|    | PATENT NO.                        | KIND | DATE     | APPLICATION NO. | DATE     |
|----|-----------------------------------|------|----------|-----------------|----------|
| PI | EP 149388                         | A1   | 19850724 | EP 1984-402584  | 19841213 |
|    | EP 149388                         | B1   | 19870812 |                 |          |
|    | R: BE, CH, DE, FR, GB, IT, LI, NL |      |          |                 |          |
|    | FR 2557105                        | A1   | 19850628 | FR 1983-20458   | 19831221 |
|    | FR 2557105                        | B1   | 19861128 |                 |          |
|    | JP 60202851                       | A2   | 19851014 | JP 1984-254262  | 19841203 |
|    | US 4595744                        | A    | 19860617 | US 1984-679571  | 19841207 |

PRAI FR 1983-20458 19831221

AB Diphenylethane **diisocyanate** (I), prepared by nitration of diphenylethane (II), hydrogenation, and **phosgenation**, is low-melting and useful in preparing polyurethanes. Thus, nitrating II with mixed acid, hydrogenating over Pd, and phosgenating gave I (2,4' 45, 4,4'

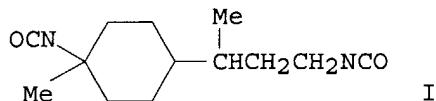
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30.3, 2,2' 15.0, 3,4' 5.0, and 2,3' 4.7%), m.p. 50°.

L9 ANSWER 25 OF 31 CAPLUS COPYRIGHT 2004 ACS on STN  
AN 1987:6494 CAPLUS  
DN 106:6494  
TI Isocyanurate-containing polyisocyanates for polyurethane coatings  
IN Halpaap, Reinhard; Klein, Gerhard; Richter, Roland; Mueller, Hanns Peter;  
Pedain, Josef; Kreuder, Hans Joachim  
PA Bayer A.-G., Fed. Rep. Ger.  
SO Ger. Offen., 37 pp.  
CODEN: GWXXBX  
DT Patent  
LA German  
FAN.CNT 1

|      | PATENT NO.      | KIND | DATE     | APPLICATION NO.                           | DATE     |
|------|-----------------|------|----------|---|----------|
| PI   | DE 3507719      | A1   | 19860911 | DE 1985-3507719                           | 19850305 |
|      | CA 1286296      | A1   | 19910716 | CA 1986-501936                            | 19860214 |
|      | EP 193828       | A1   | 19860910 | EP 1986-102373                            | 19860224 |
|      | EP 193828       | B1   | 19880427 | R: AT, BE, CH, DE, FR, GB, IT, LI, NL, SE |          |
|      | AT 33844        | E    | 19880515 | AT 1986-102373                            | 19860224 |
|      | JP 61212570     | A2   | 19860920 | JP 1986-45529                             | 19860304 |
|      | JP 06045606     | B4   | 19940615 |   |          |
|      | ES 552631       | A1   | 19880316 | ES 1986-552631                            | 19860304 |
|      | US 4851531      | A    | 19890725 | US 1987-74380                             | 19870716 |
| PRAI | DE 1985-3507719 |      | 19850305 |   |          |
|      | US 1986-831210  |      | 19860220 |   |          |
|      | EP 1986-102373  |      | 19860224 |   |          |

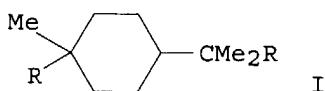
GI



AB The title compns. are prepared without oligomeric trimer formation from aliphatic-cycloaliph. diisocyanates (20-50% NCO) having 1 sterically unhindered NCO group bonded to a primary C atom and 1 sterically hindered NCO group bonded to a tertiary C atom in the ring. The **diisocyanate I** was prepared by reductive amination of 3-(1-methyl-4-cyclohexenyl)butyraldehyde, hydrocyanation, hydrolysis, and **phosgenation** of the **diamine**. Stirring I 250, crown ether KOAc complex 1.0, and PhMe 250 g at 80° for 4 h and refluxing for 15 min gave a monoisocyanurate containing 17.6% NCO and <0.3% free I.

L9 ANSWER 26 OF 31 CAPLUS COPYRIGHT 2004 ACS on STN  
AN 1984:68763 CAPLUS  
DN 100:68763  
TI Polymer intermediates from limonene  
AU Klein, Gerhard  
CS Bayer A.-G., Leverkusen, 5090, Fed. Rep. Ger.  
SO Forschungsber. - Bundesminist. Forsch. Technol., Technol. Forsch. Entwickl. (1983), BMFT-FB-T 83-053, 16 pp.  
CODEN: BFTEAJ; ISSN: 0340-7608  
DT Report  
LA German  
GI

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AB Limonene [138-86-3] was treated with HCN [74-90-8] to prepare I (R = NHCHO) [86178-67-8] which was treated with COCl<sub>2</sub> [75-44-5] to prepare I (R = isocyano) [86469-91-2]. Thermal isomerization gave I (R = cyano) [86469-91-2] which was hydrogenated to prepare I (R = CH<sub>2</sub>NH<sub>2</sub>) (II) [86469-92-3]. II was treated with COCl<sub>2</sub> to prepare I (R = CH<sub>2</sub>NCO) (III) [86469-93-4]. A film of polyamide [88666-91-5] prepared from II, isophthalic acid, and caprolactam was laminated with a polyethylene [9002-88-4] film, giving a composite film having better transparency and antiblocking properties, compared with a similar composite containing a polyamide prepared with isophoronediamine instead of II. An adduct of III and caprolactam was useful as a hardener for a saturated polyester containing OH groups. III was useful as a substitute for isophorone diisocyanate in the preparation of polyurethanes.

L9 ANSWER 27 OF 31 CAPLUS COPYRIGHT 2004 ACS on STN  
AN 1980:147777 CAPLUS

DN 92:147777

TI Aliphatic diisocyanates

IN Disteldorf, Josef; Huebel, Werner; Reiffer, Johannes; Kriebel, Guenter  
PA Chemische Werke Huels A.-G., Fed. Rep. Ger.

SO Ger. Offen., 14 pp.

CODEN: GWXXBX

DT Patent

LA German

FAN.CNT 1

|    | PATENT NO.  | KIND | DATE     | APPLICATION NO. | DATE     |
|----|-------------|------|----------|-----------------|----------|
| PI | DE 2830243  | A1   | 19800124 | DE 1978-2830243 | 19780710 |
|    | DE 2830243  | C2   | 19870716 |                 |          |
|    | JP 55033465 | A2   | 19800308 | JP 1979-86545   | 19790710 |

PRAI DE 1978-2830243 19780710

AB 1,9-Diisocyanato-5-methylnonane (I) [68882-57-5] and 1,8-diisocyanato-2,4-dimethyloctane (II) [68882-56-4] are prepared (sep. or together) by the **phosgenation** (at -10 to +40° and then at 90-180°) of the hydrogenated reaction products of isobutene and acrylonitrile. Thus, 172 g 88:12 (weight) mixture of 1,9-diamino-5-methylnonane [45024-21-3] and 1,8-diamino-2,4-dimethyloctane [51121-88-1] in 2 L PhCl was saturated at 90° with CO<sub>2</sub> and then treated at 10° with 250 g COCl<sub>2</sub> [75-44-5] in PhCl over 1.5 h. After heating the product to 80° and then 133° with addition of more COCl<sub>2</sub>, a clear solution was obtained, giving I and II.

L9 ANSWER 28 OF 31 CAPLUS COPYRIGHT 2004 ACS on STN

AN 1976:179220 CAPLUS

DN 84:179220

TI Kinetics of the reaction of crystals of toluene-2,4-diamine and 4,4'-diphenylmethanediamine dihydrochlorides with **phosgene** dissolved in chlorobenzene

AU Konstantinov, I. I.; Kormushechkina, A. I.

CS USSR

SO Zhurnal Prikladnoi Khimii (Sankt-Peterburg, Russian Federation) (1976), 49(3), 596-9

CODEN: ZPKHAB; ISSN: 0044-4618

DT Journal

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LA Russian  
AB The rate-determining step of the title reactions was the formation of the 1st-stage solid product (aminoaryl isocyanate hydrochloride) on the crystal surface. The 2nd-stage product (**diisocyanate**) then dissolved in the PhCl. The activation energies with toluene-2,4-diamine and 4,4'-diphenylmethanediamine were 12.6 and 12.0 kcal/mole, resp.

L9 ANSWER 29 OF 31 CAPLUS COPYRIGHT 2004 ACS on STN

AN 1965:67227 CAPLUS

DN 62:67227

OREF 62:11989d-f

TI Polyurethans

IN Kaplan, Melvin

PA Allied Chemical Corp.

SO 12 pp.

DT Patent

LA Unavailable

FAN.CNT 1

|    | PATENT NO. | KIND | DATE     | APPLICATION NO. | DATE |
|----|------------|------|----------|-----------------|------|
| PI | FR 1375975 |      | 19640823 | FR              |      |
|    | GB 1007785 |      |          | GB              |      |
|    | NL 297969  |      |          | NL              |      |
|    | US 3215652 |      | 1965     | US              |      |

PRAI US 19620924

AB Polyether-polyols are treated with tolylene **diamine** (I) **phosgenation** products (amine equivalent 98-120), NCO-OH equivalent ratio 1.03: 1-1.05: 1, in the presence of a mixture of a blowing agent, tertiary amine, organotin compound, and a siloxane-oxyalkylene block copolymer to give rigid foams which are weak heat conductors. Thus, a solution (90°) of 244 parts I in 880 parts PhCl is added to a mixture (-10°) of 560 parts COCl<sub>2</sub> and 120 parts PhCl at a maximum of 20°. The mixture is heated to 136°, a HCl-COCl<sub>2</sub>-PhCl mixture is distilled, PhCl is distilled at 25 mm., and the residue is distilled at 10-12 mm. to give 70 parts tolylene **diisocyanate** (II) and 280 parts residue (III) (amine equivalent 106.3, viscosity (25°) 61 cp., d<sub>25</sub> 1.26) containing 74% II. A mixture of Actol 52-460 (OH number >460) 100, Bu<sub>2</sub>Sn dilaurate 0.37, Me<sub>2</sub>NCH<sub>2</sub>CH<sub>2</sub>OH 1.0, Dow Corning 113 fluid 1.5, and FCCl<sub>3</sub> 38 parts is introduced into a mixer at 5.5 kg./min. at 23°, as III is introduced at 3.6 kg./min. at 22°, to give a mixture, NCO-OH ratio 1.03, which becomes hard after 6.5 min.; the product is molded at 41° to give a panel, d. 29 kg./m.<sup>3</sup>, containing 96.9% closed cells, and having a compression resistance of 1.3 kg./cm.<sup>2</sup> and a heat conductivity of 0.258 kcal./hr./m<sup>2</sup>/cm./°C.

L9 ANSWER 30 OF 31 CAPLUS COPYRIGHT 2004 ACS on STN

AN 1963:441342 CAPLUS

DN 59:41342

OREF 59:7403d-f

TI Preparation of tolylene **diisocyanate**. II. **Phosgenation** of tolylenediamine

AU Murakami, Tomohisa

CS Hodogaya Chem. Ind. Co., Yokohama, Japan

SO Yuki Gosei Kagaku Kyokaishi (1963), 21, 458-62

CODEN: YGKKAЕ; ISSN: 0037-9980

DT Journal

LA Unavailable

AB Reaction of diamines and COCl<sub>2</sub> at low temperature for conversion of the amino groups into a carbamoyl chloride and an amine-HCl group, and further reaction at high temperature with COCl<sub>2</sub> is known. When a **diamine** such

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as tolylenediamine (I) is used, a high mol. weight urea compound is formed under the wrong reaction conditions and precipitates, so that the reaction with COCl<sub>2</sub> becomes difficult, resulting in a considerable decrease in the reaction velocity and yield. For successful operation of this reaction, it is necessary to make sufficient contact of COCl<sub>2</sub> with I in the low temperature reaction in order to decrease the amount of unreacted I in the product

and give a sufficient supply of COCl<sub>2</sub> in the high temperature reaction. It is desirable to use good solvents (e.g. ketone, esters, ethers) for the low temperature reaction product. PhCl, C<sub>6</sub>H<sub>4</sub>Cl<sub>2</sub>, etc., could be used as solvents by

adding I solution to COCl<sub>2</sub> solution with stirring, in order to give finely dispersed particles of the low temperature reaction product. The high temperature

reaction is completed within 30 min. at 130° by sufficient introduction of COCl<sub>2</sub> to the low temperature reaction product and the yield is high.

L9 ANSWER 31 OF 31 CAPLUS COPYRIGHT 2004 ACS on STN

AN 1950:679 CAPLUS

DN 44:679

OREF 44:109h-i, 110b-i, 111a-i, 112a-i, 113a-i, 114a-i, 115a-b

TI Polyurethans. IV. Mono- and polyisocyanates

AU Sieffken, Werner

SO Ann. (1949), 562, 75-136

DT Journal

LA Unavailable

OS CASREACT 44:679

GI For diagram(s), see printed CA Issue.

AB cf. Bayer, C.A. 42, 6160c; Hebermehl, C.A. 43, 1604i. A comprehensive review of the researches in this field made by the staff of the former I. G. Farbenind., W. Altner, D. Delfs, A. Dierichs, E. Hartmann, E. Liese, A. Modersohn, S. Petersen, E. Prell, R. Putter, H. Rinke, W. Schulte, G. Schwaebel, H. Schwarz, G. Spielberger, K. Taube, A. Pielmann, K. Sigwart, H. Brock, J. Mierbach, E. Scholz, H. Glaser, F. Moller, and R. Schroter, including a brief literature survey with 62 refs. In general the HCl salts of amines were treated with COCl<sub>2</sub> in excess, and the resultant HCl was rapidly removed by choosing a solvent in which the RNCO, but not HCl was soluble. Temps. were regulated to insure conversion of the intermediate RNHCOCl into RNCO. In the aliphatic series, the yields of RNCO were uniformly satisfactory and arylaliph. amines, or alicyclic or heterocyclic amines, underwent very similar conversions. The reaction also applied to compds. of the type RCH(NH<sub>2</sub>)R'. Solvents used included PhMe, xylene, PhCl, Cl<sub>2</sub>C<sub>6</sub>H<sub>4</sub>, and C<sub>6</sub>H<sub>3</sub>Cl<sub>3</sub>, so chosen that the b.ps. of the solvent and the resulting RNCO showed a sufficient difference. The amine-HCl may be dissolved in the solvent, or the free amine may be dissolved and then treated with dry HCl. Normally COCl<sub>2</sub> was added until any insol. HCl salt was fully dissolved, the volatile gases then swept out by means of an inert gas, and the resulting products fractionated. Polymerization products

were

retained as still residues; their amts. could be materially increased by the use of metallic catalysts (such as FeCl<sub>3</sub>, SnCl<sub>4</sub>, ZnCl<sub>2</sub>, Fe carbonyl derivs., etc.). In individual cases, HCl is removed from RNHCOCl by means of Ca(OH)<sub>2</sub> or derivs. of CH<sub>2</sub>.O.CH<sub>2</sub> (sometimes resulting in decreased yields of RNCO). In special cases were used substituted ureas of the type RR'NCONHR'' (formed from RR'NCOC<sub>2</sub> + H<sub>2</sub>NR''), where R'' is a relatively small alkyl group and may be converted into R''NCO by heating above 200°. Another possibility was to treat compds. of the type 2-HOC<sub>6</sub>H<sub>4</sub>OCONHR with the formation of RNCO and o-C<sub>6</sub>H<sub>4</sub>(OH)<sub>2</sub>. The conversions of amines containing such substituents as Cl, CN, OR', CO<sub>2</sub>R', COCl, etc., into analogous isocyanates is discussed. Diamine, triamine, or tetramine HCl salts on COCl<sub>2</sub> treatment may be converted into

analogous di-, tri-, and tetraisocyanates. Difficulties in the choice of suitable (large-scale) reactors are discussed. Whereas for an aliphatic RNCO, **phosgenation** may be carried out in stainless steel, aromatic di- and triamines require Pb-lined reactors. Batch or continuous **phosgenation** may be used. In determining the percentage NCO in an isocyanate, 2 methods were used: a cumbersome gravimetric method depending on the formation of a difficultly soluble Ph urea by condensation of RNCO with PhNH<sub>2</sub>, and the more practical procedure in which RNCO is treated with a known excess of Bu<sub>2</sub>NH in PhCl, thus giving rise very rapidly to Bu<sub>2</sub>NCONHR, and the unchanged Bu<sub>2</sub>NH is then titrated with HCl in the presence of MeOH (within 5 min. after inception of the quant. reaction). In the formation of 1,6-hexamethylene **diisocyanate** (I), b15 132° [bis(methylurethane), m. 113-14°], on repeated fractionation a small forerun was 6-chlorohexyl isocyanate (II), b12 108° [methylurethane (III), b10 150-2°; Cl(CH<sub>2</sub>)<sub>6</sub>NHCONH<sub>2</sub>, m. 128-9°]. [HCl.H<sub>2</sub>N(CH<sub>2</sub>)<sub>3</sub>]<sub>2</sub> loses NH<sub>4</sub>Cl, forming CH<sub>2</sub>.(CH<sub>2</sub>)<sub>4</sub>.CH<sub>2</sub>.NH.HCl, which reacts with COCl<sub>2</sub> to give CH<sub>2</sub>.(CH<sub>2</sub>)<sub>4</sub>.CH<sub>2</sub>.NCOCl (isomeric with II), b11 116-18° (methylurethane, C<sub>6</sub>H<sub>12</sub>NCO<sub>2</sub>Me, b13 96-7°; and urea, C<sub>6</sub>H<sub>12</sub>NCONH<sub>2</sub>, m. 123-4°). III heated with PhONa formed PhOCH<sub>2</sub>(CH<sub>2</sub>)<sub>5</sub>NHCO<sub>2</sub>Me, which when heated with aqueous HCl-AcOH gave 6-phenoxyhexylamine-HCl, m. 142-3°. I in pseudocumene, heated 2 h. with pure COCl<sub>2</sub> at 160-65°, was not converted into II. On the other hand mixts. of HCl (gas) and COCl<sub>2</sub> acting on I gave small amts. of II after 48 h. **phosgenation**. The course of the reaction is discussed. Possibly Cl<sub>2</sub>CN(CH<sub>2</sub>)<sub>6</sub>NCO is first formed from I and then split into ClCN and II. MeO(CH<sub>2</sub>)<sub>3</sub>NH<sub>2</sub>.HCl treated 7 h. in 1-C<sub>10</sub>H<sub>7</sub>Cl at 140-50° with COCl<sub>2</sub> with stirring gave 81% Cl(CH<sub>2</sub>)<sub>3</sub>NCO, b16 54.6-4.8°, together with smaller amts. of (ClCH<sub>2</sub>)<sub>2</sub>CH<sub>2</sub>, b1630°, which with PhONa yielded (PhOCH<sub>2</sub>)<sub>2</sub>CH<sub>2</sub>, m. 61° (from EtOH). NC(CH<sub>2</sub>)<sub>5</sub>NH<sub>2</sub>, b16 118-19°, treated in PhCl with HCl, followed by COCl<sub>2</sub> at 120-30°, gave 94% NC(CH<sub>2</sub>)<sub>5</sub>NCO, b12 134-5° [NC(CH<sub>2</sub>)<sub>5</sub>NHCONH<sub>2</sub>, m. 142° (from EtOH)]. CH<sub>2</sub>(NH<sub>2</sub>.HCl)CO<sub>2</sub>Et in PhMe with COCl<sub>2</sub> gave 84.5% OCNCH<sub>2</sub>CO<sub>2</sub>Et, b11 67-8°, which with PhNH<sub>2</sub> in Et<sub>2</sub>O yielded PhNHCONHCH<sub>2</sub>CO<sub>2</sub>Et, m. 111° (from EtOH). From BuO(CH<sub>2</sub>)<sub>3</sub>NH<sub>2</sub>.HCl in PhMe was formed BuO(CH<sub>2</sub>)<sub>3</sub>NCO, b11 76-8°, and PhNHCONH(CH<sub>2</sub>)<sub>3</sub>OBu, m. 59-60° (from Et<sub>2</sub>O). By heating Ph<sub>2</sub>NCONHMe at 240-90°, 1 mol. MeNCO, b. 38-40°, and 1 mol. Ph<sub>2</sub>NH were formed. 2-HOC<sub>6</sub>H<sub>4</sub>O<sub>2</sub>CNHEt, m. 142-4°, at 210-50° yielded EtNCO, b. 58-60°, and O-(HO)C<sub>6</sub>H<sub>4</sub>. In 1-C<sub>10</sub>H<sub>7</sub>Cl at 150° (PhNH)<sub>2</sub>CO with COCl<sub>2</sub> gave PhNCO, b16 55-7°. In C<sub>6</sub>H<sub>4</sub>Cl<sub>2</sub>, 3-HOCH<sub>2</sub>-C<sub>6</sub>H<sub>4</sub>NH<sub>2</sub>.HCl and COCl<sub>2</sub> gave 3-ClCH<sub>2</sub>C<sub>6</sub>H<sub>4</sub>NCO (V), b11 118-19°; 3-ClCH<sub>2</sub>C<sub>6</sub>H<sub>4</sub>NHCO<sub>2</sub>Me, m. 87-8°, with pyridine yields 1-[3-(carbomethoxyamino)benzyl]pyridinium chloride, C<sub>14</sub>H<sub>15</sub>O<sub>2</sub>N<sub>2</sub>Cl, m. 223-4°. Similarly, 3-MeOCH<sub>2</sub>C<sub>6</sub>H<sub>4</sub>NH<sub>2</sub>.HCl (VI) on **phosgenation** gave 96% 3-MeOCH<sub>2</sub>C<sub>6</sub>H<sub>4</sub>NCO, b11 108°, and 4% V; 3-MeOCH<sub>2</sub>-C<sub>6</sub>H<sub>4</sub>NHCONH<sub>2</sub>, m. 122-4°. VI treated in C<sub>6</sub>H<sub>4</sub>Cl<sub>2</sub> at 140° with HCl prior to **phosgenation** gives 3-ClCH<sub>2</sub>C<sub>6</sub>H<sub>4</sub>-NH<sub>2</sub>.HCl, converted readily into V. 3,3'-Bianisidine HCl salt in PhCl and COCl<sub>2</sub> at 150-60° gave after vacuum distillation at 150° a high yield of 3,3'-dimethoxy-4,4'-biphenylene **diisocyanate** (VII), m. 121-2°, forming a complex, 2VII.PhCl, m. 125-6°. The bis(methylurethane) of VII m. 215-16°. The following are examples of **phosgenation** of free bases. When 800 g. COCl<sub>2</sub> in 2 l. O-Cl<sub>2</sub>C<sub>6</sub>H<sub>4</sub> in an ice-salt bath was treated with 200 g. molten (4-H<sub>2</sub>NC<sub>6</sub>H<sub>4</sub>)<sub>2</sub>CH<sub>2</sub> so that a temperature of about 0° was maintained, and the suspension was then heated to 130°, 215 g. (4-OCNC<sub>6</sub>H<sub>4</sub>)<sub>2</sub>CH<sub>2</sub>, b0.1 156-8°, was formed [bis(methylurethane), C<sub>17</sub>H<sub>18</sub>O<sub>4</sub>N<sub>2</sub>, m. 183-3.5°]. Similarly in PhCl, 4-H<sub>2</sub>NC<sub>6</sub>H<sub>4</sub>CO<sub>2</sub>Me with COCl<sub>2</sub> gave 4-OCNC<sub>6</sub>H<sub>4</sub>CO<sub>2</sub>Me, b11 122-4°, m. 49° (methylurethane, m. 177-8°), and tetrahydro-1,5-naphthylenediamine in C<sub>6</sub>H<sub>4</sub>Cl<sub>2</sub> gave a mixture of tetrahydro-1,5-naphthylenene **diisocyanate**, b0.09 129-32°, and a smaller amount of

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7,8-dihydro-1-naphthyl isocyanate, b15 137-8° [methylurethane, m. 86-7°; phenylurea (VIII), m. 210-12°], which was shown not to be identical with 1-naphthyl isocyanate, b12 140-2° (methylurethane, m. 122-3°; phenylurea, m. 220-2°), or with tetrahydro-ar-1-naphthyl isocyanate, b14 134-5° [methylurethane, m. 62-3°; phenylurea, m. 193-4°, also formed by hydrogenating VIII]. The following are examples of **phosgenation** of carbamic acids. 1,4-Diaminocyclohexane (345 g.) in 3 l. o-C<sub>12</sub>C<sub>6</sub>H<sub>4</sub> was saturated at 90-95° with CO<sub>2</sub>, stirred 8 h., 700 g. COCl<sub>2</sub> introduced at 0°, the CO<sub>2</sub> removed, the mixture heated to 160°, and more COCl<sub>2</sub> added until after 14-16 h. the solution was clear; fractional distillation

yielded a mixture of 1,4-cyclohexane diisocyanates, 1,4-(ONC)<sub>2</sub>C<sub>6</sub>H<sub>10</sub>, transform, m. 63-4° (from petr. ether) [bis(methylurethane), m. 264° (from MeOH)], and liquid cis form [characterized by its bis-(methylurethane), C<sub>10</sub>H<sub>18</sub>O<sub>4</sub>N<sub>2</sub>, m. 139-40° (from Me<sub>2</sub>CO)]. Similarly, 4,4'-diaminodicyclohexylmethane gave 4,4'-(dicyclohexyl)methane **diisocyanate**, (4-OCNC<sub>6</sub>H<sub>10</sub>)<sub>2</sub>CH<sub>2</sub>, salvelike mass, b0.5-0.6 165-80°. The following are examples of **phosgenation** in the vapor phase. A mixture of 450 g. COCl<sub>2</sub> and 245 g. PhNH<sub>2</sub> passed in 1 h. through a tube at 230-40° while 130 g. 1-C<sub>10</sub>H<sub>7</sub>Cl was dropped in, gave 86% PhNCO. By entraining 80 g. p-(H<sub>2</sub>N)<sub>2</sub>C<sub>6</sub>H<sub>4</sub> with 35 l. CO<sub>2</sub> at 270-80° per h., heating the mixture to 470°, introducing 470 g. COCl<sub>2</sub>/h., and condensing in PhCl, S. obtained p-C<sub>6</sub>H<sub>4</sub>(NCO)<sub>2</sub>, m. 93-4° (after sublimation) [bis(methylurethane), m. 207°].

To 86.7 g. 3-HO<sub>2</sub>CC<sub>6</sub>H<sub>4</sub>NH<sub>2</sub>.HCl in 2 l. H<sub>2</sub>O containing 30 cc. HCl was added 42 cc. CS<sub>2</sub>Cl<sub>2</sub>, thus forming 67 g. 3-HO<sub>2</sub>CC<sub>6</sub>H<sub>4</sub>NCS, m. 165° (from MeOH); acid chloride, b14 152-4°, m. 22-3°, 40 g. of which in 200 cc. PhCl refluxed with 16 g. powdered NaN<sub>3</sub> gave 32 g. 3-OCNC<sub>6</sub>H<sub>4</sub>CNS, b14 140-2°, f.p. 4-6°. The following other isocyanates, RNCO, and their derivs. were prepared by methods analogous to those outlined. Most of these are new, but no differentiation has been made between new compds. and products previously prepared. In all cases (uncor.) b.p. or m.ps. were checked and some of the compds. were obviously impure. R = CH<sub>2</sub>:CHCH<sub>2</sub>, b. 87-9°; Pr, b. 88°; Bu, b. 114-16°

(corresponding phenylurea, m. 129-30°); Me<sub>2</sub>CHCH<sub>2</sub>, b. 104-5° (phenylurea, m. 151-2°); Me<sub>3</sub>C, b. 85° (phenylurea, m. 167-8°). Am, b. 136-7° (AmNHCONHPh, m. 92-3°); isohexyl, b14 46°; BuCHEtCH<sub>2</sub>, b15 75-9°; dodecyl, b11 140-6°; tetradecyl, b14 165-70°; hexadecyl, b14 186-8°; oleyl, b0.05-0.06 135-40°; octadecyl, b11 190-210°; CH<sub>2</sub>.CH<sub>2</sub>.SO<sub>2</sub>.CH<sub>2</sub>.CH (not characterized) (phenylurea, m. 190°); cyclohexyl, b11 54°; 2-decahydronaphthyl, b12 116-17°; C<sub>6</sub>H<sub>11</sub>CH<sub>2</sub>C<sub>6</sub>H<sub>10</sub>, b0.25 120-3°; MeEtCH, b. 101-2° (phenylurea, m. 154°); iso-PrCHMe, b. 118-20° (phenylurea, m. 143-4°); Et<sub>2</sub>CH, b. 125-6° (phenylurea, m. 177-8°); Pr<sub>2</sub>CH, b13 58-9°; PhCHMe, b13 90-4°; PhCH<sub>2</sub>CH<sub>2</sub>CHMe, b12 117°; (C<sub>6</sub>H<sub>13</sub>)<sub>2</sub>CH, b12 139-41°; PrCH(C<sub>11</sub>H<sub>23</sub>), b12 150-70°; C<sub>17</sub>H<sub>35</sub>CHMe, b0.1 140-60°; ClCH<sub>2</sub>CH<sub>2</sub>, b16 42° [(ClCH<sub>2</sub>CH<sub>2</sub>NH)<sub>2</sub>CO, m. 128-9°]; Cl(CH<sub>2</sub>)<sub>4</sub>, b11 70-1° (phenylurea, m. 107°); Cl(CH<sub>2</sub>)<sub>5</sub>, b13 100° (methylurethane, m. 44°); Cl(CH<sub>2</sub>)<sub>7</sub>, b13 111°; Cl(CH<sub>2</sub>)<sub>4</sub>O(CH<sub>2</sub>)<sub>2</sub>CH<sub>2</sub>, b0.1 76°; NC(CH<sub>2</sub>)<sub>2</sub>, b12 104°; [NC(CH<sub>2</sub>)<sub>2</sub>NH<sub>2</sub>CO, m. 148°]; NC(CH<sub>2</sub>)<sub>3</sub>, b10 103°; [NC(CH<sub>2</sub>)<sub>3</sub>NHCONH<sub>2</sub>, m. 142°]; MeO<sub>2</sub>C(CH<sub>2</sub>)<sub>5</sub>, b14 118°; MeO(CH<sub>2</sub>)<sub>3</sub> (impure) (phenylurea, m. 79-80°); EtO(CH<sub>2</sub>)<sub>3</sub> (impure) (phenylurea, m. 66°); PrO(CH<sub>2</sub>)<sub>3</sub>, b11 60-1° (phenylurea, m. 62°); iso-Pr(CH<sub>2</sub>)<sub>3</sub> (impure); BuO(CH<sub>2</sub>)<sub>3</sub>, b11 76-8° (phenylurea, m. 59-60°); iso-BuO(CH<sub>2</sub>)<sub>3</sub>, b14 78° (phenylurea, m. 80°); sec-BuO(CH<sub>2</sub>)<sub>3</sub> (impure) (phenylurea, m. 53-4°); cyclohexyloxypropyl, b11 113-14° (phenylurea, m. 100°); BuOCH<sub>2</sub>CH<sub>2</sub>O(CH<sub>2</sub>)<sub>3</sub>, b11 123-4° (phenylurea, liquid); C<sub>8</sub>H<sub>17</sub>O(CH<sub>2</sub>)<sub>3</sub>, b11

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135° (phenylurea, m. 55-6°); iso-C<sub>8</sub>H<sub>17</sub>, isomer, b11  
125-8°; C<sub>10</sub>H<sub>21</sub>O(CH<sub>2</sub>)<sub>3</sub>, b11 160-2° (phenylurea, m.  
58-9°); 3-(iso-octylcyclohexyloxy)propyl, b0.1-0.2 140-52°;  
o-tolyl, b9.5 63.2-3.4°; m-tolyl, b9.5 65.7-66.3°;  
p-tolyl, b10 67.6-67.8°; benzyl, b10 82-4° (phenylurea, m.  
167-8°); xylyl (tech.), b12 78-80°; phenethyl, b10  
98-100° (urea, m. 112°); 2,4,5-Me<sub>3</sub>C<sub>6</sub>H<sub>2</sub>, b12 100-1°;  
2,4,6-isomer, b11 96-7°; Ph(CH<sub>2</sub>)<sub>3</sub>, b12 115°;  
4-cyclohexylphenyl, b4 128-30°; 4-PhCH<sub>2</sub>C<sub>6</sub>H<sub>4</sub>, b0.07 120°  
(methylurethane, m. 78-9°; urea, m. 158-9°);  
2-methyl-4-cyclohexylphenyl, b4.5 138-42°; 3,4,6-Cl<sub>3</sub>C<sub>6</sub>H<sub>2</sub>, b11  
124-6°, m. 60-1°; 3, 4-Cl<sub>2</sub>C<sub>6</sub>H<sub>3</sub>, b12 111-12°, m.  
45° (methylurethane, m. 111-12°; urea, m. 153-4°);  
2-O<sub>2</sub>NC<sub>6</sub>H<sub>4</sub>, b17 135°, m. 40-1°; 3-isomer, b11 130-1°,  
m. 50-1°; 4-isomer, b11 137-8°, m. 57°; 2-ClC<sub>6</sub>H<sub>4</sub>,  
b10 83.5°; 3-isomer, b10.5 83-6°; 4-isomer, b9.5  
80.6-0.9°, m. 31-2° (methylurethane, m. 115°);  
3-FO<sub>2</sub>SC<sub>6</sub>H<sub>4</sub>, b31-2 154-6°, m. 25° (methylurethane, m.  
93°); 3-NCC<sub>6</sub>H<sub>4</sub>, b12 123°, m. 50-1° (methylurethane,  
m. 97-8°); 4,3-Cl(F<sub>3</sub>C)C<sub>6</sub>H<sub>3</sub>, b14 86-9°; 3,4-HO<sub>2</sub>C(HO)C<sub>6</sub>H<sub>3</sub>,  
decompose (methylurethane, m. 221°); 2, 4-Me(O<sub>2</sub>N)C<sub>6</sub>H<sub>3</sub>, b23  
168°, m. 75-8°; 4, 3-Me-(O<sub>2</sub>N)C<sub>6</sub>H<sub>3</sub>, b0.2 100-2°;  
2-MeOC<sub>6</sub>H<sub>4</sub>, b10 94.8-5.5°; 3-isomer, b10 94.6-4.8°;  
4-isomer, b9.5 47.6-7.8°; 2, 5-Me(SCN)C<sub>6</sub>H<sub>3</sub>, b16.5 164°  
(methylurethane, m. 136-7°); 4,3-Cl(MeO<sub>2</sub>CNH)C<sub>6</sub>H<sub>3</sub>, b11 116  
°, m. 80°; 4-MeO<sub>2</sub>CC<sub>6</sub>H<sub>4</sub>, b. 122-4°, m. 49°  
(methylurethane, m. 177-8°); 2,4,5-Me<sub>2</sub>(O<sub>2</sub>N)C<sub>6</sub>H<sub>2</sub>, b2 110-25°;  
3-MeCHClC<sub>6</sub>H<sub>4</sub>, b11 116°; EtOC<sub>6</sub>H<sub>4</sub>, b12 104-6°; 2,  
5-(MeO)<sub>2</sub>C<sub>6</sub>H<sub>3</sub>, b26-7 152-4°; 4-EtO<sub>2</sub>CC<sub>6</sub>H<sub>4</sub>, b17 144-8°, m.  
30-2°; 3, 4-Cl<sub>2</sub>C<sub>6</sub>H<sub>3</sub>(CH<sub>2</sub>)<sub>3</sub>, b14-15 176-8°; 4,3-Me(MeO<sub>2</sub>CNH)  
C<sub>6</sub>H<sub>3</sub>, m. 87°; 4,3-Me(EtO<sub>2</sub>CNH)C<sub>6</sub>H<sub>3</sub>, m. 72°; 4,  
2-Cl(4-ClC<sub>6</sub>H<sub>4</sub>O)C<sub>6</sub>H<sub>3</sub>, m. 44-6°; 3-PhMeNSO<sub>2</sub>C<sub>6</sub>H<sub>4</sub>, m. 74-5°;  
4-PhN:NC<sub>6</sub>H<sub>4</sub>, m. 97-8° (ethylurethane, m. 151°); 3,  
4-Me(4-MeOC<sub>6</sub>H<sub>4</sub>N:N)C<sub>6</sub>H<sub>3</sub>, m. 80°; 5-methyl-2-methoxy-4-(2-  
chlorophenylazo)phenyl, m. 153°; 2-naphthyl, b14 144°, m.  
56° (methylurethane, m. 113°); 5-nitro-1-naphthyl, m.  
121-2° (methylurethane, m. 170°); 8-chloro-1-naphthyl, b0.1  
125-7° (methylurethane, m. 119-20°); 1-chloro-2-naphthyl,  
b0.3 120°, m. 52-3° (methylurethane, m. 115-16°);  
tetrahydro-ac-2-naphthyl, b10 134-6° (phenylurea, m.  
169-70°); decahydro-2-naphthyl, b12 116-17°; 3-phenanthryl,  
b4 198-204°, m. 48° (methylurethane, m. 140-2°);  
4-(6-methyl-2-benzothiazolyl)phenyl, m. 143-4° (methylurethane, m.  
203-5°); 9-ethyl-3-carbazolyl, b0.1 172-8°, m. 48°  
(methylurethane, m. 118-20°); 3-(9-carbazolyl)propyl, b7  
240-2° (methylurethane, m. 107-9°); 3-pyrenyl, m. 92°  
(methylurethane, m. 203°); 12-chrysanyl, m. 155-6°  
(methylurethane, m. 204°); 9,10-ethanoanthr-11-ylmethyl, m.  
92-4° (methylurethane, m.p. not given). The following  
diisocyanates (and derivs.) were prepared, a number of them by an azide  
degradation

method not discussed (R = -NCO): (CH<sub>2</sub>CH<sub>2</sub>R)<sub>2</sub>, b14 75-6°; R(CH<sub>2</sub>)<sub>3</sub>R,  
b14 86-7°; RCH<sub>2</sub>CH:CHCH<sub>2</sub>R (impure) [bis-(methylurethane), m.  
137°]; R(CH<sub>2</sub>)<sub>4</sub>R, b14 102-4° [bis-(methylurethane), m.  
129-30°]; (RCH<sub>2</sub>CH<sub>2</sub>)<sub>2</sub>S, b0.5-1 100-20° {bis-  
urea[H<sub>2</sub>NCONH(CH<sub>2</sub>)<sub>2</sub>]<sub>2</sub>S, m. 210°}; R(CH<sub>2</sub>)<sub>5</sub>R, b15 123-5°  
[bis(methylurethane), m. 114-15°]; RCH<sub>2</sub>CHMeCH<sub>2</sub>CH<sub>2</sub>R, b9 98-9°  
(bis-urea, m. 181-2°); R(CH<sub>2</sub>)<sub>6</sub>R, b14 130-2°  
[bis(methylurethane), m. 113-14°]; [RCH<sub>2</sub>(CH<sub>2</sub>)<sub>2</sub>]<sub>2</sub>O, b14 137°  
(bis-urea, m. 141°); S-analog, b0.3-0.4 118°; R(CH<sub>2</sub>)<sub>7</sub>R, b14  
140-2° [bis(methylurethane), m. 97.5-8°]; RCH<sub>2</sub>CMe<sub>2</sub>(CH<sub>2</sub>)<sub>2</sub>H<sub>2</sub>R,  
b12 120-2°, b16 132-4°; RCH<sub>2</sub>(CH<sub>2</sub>)<sub>2</sub>CH(OMe)(CH<sub>2</sub>)<sub>2</sub> CH<sub>2</sub>R, b12

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142-5°, (p-chlorophenylurea, m. 217-18°); R(CH<sub>2</sub>)<sub>8</sub>R, b11  
146-8° [bis(methylurethane), m. 111-12°];  
RCH<sub>2</sub>CMe<sub>2</sub>CH<sub>2</sub>CHMeCH<sub>2</sub>R, b5 106° (bis-urea, m. 159-62°);  
R(CH<sub>2</sub>)<sub>9</sub>R, b0.5 121° [bis(methylurethane), m. 102-5°];  
R(CH<sub>2</sub>)<sub>10</sub>R, b0.8 128° [bis(methylurethane), m. 115°];  
R(CH<sub>2</sub>)<sub>2</sub>CH(OBu)(CH<sub>2</sub>)<sub>3</sub>R, b2 142-55° (decomposition);  
R(CH<sub>2</sub>)<sub>3</sub>O(CH<sub>2</sub>)<sub>4</sub>O(CH<sub>2</sub>)<sub>3</sub>R, b0.1 130-5°; R(CH<sub>2</sub>)<sub>11</sub>R, 0.06 124°  
[bis(methylurethane), m. 97-8°]; R(CH<sub>2</sub>)<sub>12</sub>R, b0.08 135°  
[bis(methylurethane), m. 117-18°]; [R(CH<sub>2</sub>)<sub>6</sub>]<sub>2</sub>S, b0.7-0.8  
180-5° [bis(methylurethane), m. 87°]; m-C<sub>6</sub>H<sub>4</sub>(CH<sub>2</sub>R)<sub>2</sub>, b12  
159-62°, [bis(methylurethane), m. 110-11°]; p-isomer, b16  
172°, m. 45-6° [bis(methylurethane), m. 184°]; 1,  
2-C<sub>6</sub>H<sub>10</sub>(CH<sub>2</sub>R)<sub>2</sub> (cyclohexane), b23-24 165-8°; 1,4-isomer, b11  
154-6° [bis(methylurethane), m. 163°]; p-C<sub>6</sub>H<sub>4</sub>(CH<sub>2</sub>CH<sub>2</sub>R)<sub>2</sub>,  
b0.1-0.2 142-5°; 1, 4-C<sub>10</sub>H<sub>6</sub>(CH<sub>2</sub>R)<sub>2</sub>, b1 183-4°  
[bis(methylurethane), m. 167°]; 1, 5-isomer, m. 88-9°  
[bis(methylurethane), m. 215-16°]; RCH<sub>2</sub>CH.CH<sub>2</sub>.CHMe.CH<sub>2</sub>.CHMe.CH(CH<sub>2</sub>)  
3R, b4 165-72°; [C<sub>6</sub>H<sub>4</sub>(CH<sub>2</sub>)<sub>3</sub>R]<sub>2</sub>, m. 29° [bis(methylurethane),  
m. 156-7°]; (IX), m. 235-7° (analogous dicyanate not  
characterized). RCH.CH<sub>2</sub>.CHR.CH<sub>2</sub>.CH<sub>2</sub>.CH<sub>2</sub>, b14 120-30°; impure  
CHMe.CHR.CH<sub>2</sub>.CHR.CH<sub>2</sub>.CH<sub>2</sub>, b12 127-9° Et homolog, b19 140-4°;  
CH<sub>2</sub>(CH<sub>2</sub>.CH<sub>2</sub>.CHR.CH<sub>2</sub>.CH<sub>2</sub>.CH)<sub>2</sub>, b0.5-0.6 165-80°; MeCH(C<sub>6</sub>H<sub>10</sub>R)<sub>2</sub>  
homolog, b4.5 198-208° [bis-(methylurethane), m. 182-3°];  
Me<sub>2</sub>C(C<sub>6</sub>H<sub>10</sub>R)<sub>2</sub> homolog, b8-10 208-30°; (CH<sub>2</sub>.CH<sub>2</sub>.CHR.CH<sub>2</sub>.CHMe.CH)<sub>2</sub>  
CH<sub>2</sub>, b5 200-12°; 3,3'-dimethyl isomer, b9 198-210°;  
3,3',5,5'-tetra-Me homolog, b7 205-12° [bis(methylurethane), m.  
204-7°]; p-RCH<sub>2</sub>C<sub>6</sub>H<sub>4</sub>R, b19 152°; p-RCH<sub>2</sub>CH<sub>2</sub>C<sub>6</sub>H<sub>4</sub>R, b15  
156°; m-RCH(Me)C<sub>6</sub>H<sub>4</sub>R, b11 134° [bis(methylurethane),  
m. 112-13°]; m-RCH<sub>2</sub>CH<sub>2</sub>CH<sub>2</sub>C<sub>6</sub>H<sub>4</sub>R, b0.5 118-20°; p-isomer,  
b0.5 100-115°; m-RCHMeCH<sub>2</sub>CH<sub>2</sub>C<sub>6</sub>H<sub>4</sub>R, b2 146-9°; p-isomer,  
b3.5 142-5°; tetrahydro-1,5-naphthylene diisocyanate,  
b0.04 117-18°, 4-(4-RC<sub>6</sub>H<sub>10</sub>)C<sub>6</sub>H<sub>4</sub>R (termed "4,4'-hexahydrobenzidine  
derivative"), b0.7 155-60°, m. 38-44° [bis(methylurethane), m.  
216° (poorly)]; 4-(4-RC<sub>6</sub>H<sub>10</sub>-CH<sub>2</sub>)C<sub>6</sub>H<sub>4</sub>R, b0.2-0.3, m. 152-6°;  
m-C<sub>6</sub>H<sub>4</sub>R<sub>2</sub> b12, 104-6°, m. 51° [bis(methylurethane), m.  
157-7.5°]; p-isomer, b12 110-12°, m. 94-6°  
[bis(methylurethane), m. 209-10°]; 1,2,4-MeC<sub>6</sub>H<sub>3</sub>R<sub>2</sub>, b18  
124-6°, m. 21° (33° also given) [bis(methylurethane),  
m. 170-1°]; 1,2,6-isomer, b18 129-33°; 1,2,5-isomer, b15  
138-9°, m. 39°; 1,3,5-isomer, b17 133.5-4.5°;  
1,3,2,4-Me<sub>2</sub>C<sub>6</sub>H<sub>2</sub>R<sub>2</sub>, b17 140-4°; 1,3,4,6-isomer, b15 139°, m.  
70-1°; 1,4,2,5-isomer, b14 138-43°, m. 82° (also  
given as 76°); 1,2,4-EtC<sub>6</sub>H<sub>3</sub>R<sub>2</sub>, b12 124-6°;  
1,2,4-iso-PrC<sub>6</sub>H<sub>3</sub>R<sub>2</sub>, b12 136-40° [bis(methylurethane), m.  
160-7°]; ??-Et<sub>2</sub>C<sub>6</sub>H<sub>2</sub>R<sub>2</sub> b11 138-40°; ??-iso-Pr<sub>2</sub>C<sub>6</sub>H<sub>2</sub>R<sub>2</sub>, b12  
148-60°; 1,2,4-ClC<sub>6</sub>H<sub>3</sub>R<sub>2</sub>, b11 122-4°; 1,2,4-O<sub>2</sub>NC<sub>6</sub>H<sub>3</sub>R<sub>2</sub>,  
b14 160-80° [bis(methylurethane), m. 197-8°]; 1,2,5-isomer,  
b4 165-73°, m. 59-61° [bis(methylurethane), m.  
157-8°]; 1,3,4,6-C<sub>12</sub>C<sub>6</sub>H<sub>2</sub>R<sub>2</sub>, b12 140-6°; 1,3,2,4-isomer,  
b1.5 120-6°, m. 64-5° [bis(methylurethane), m.  
184-5°]; 1,4,2,5-isomer, m. 134-7°;  
1,4,2,5-Cl(MeO)C<sub>6</sub>H<sub>2</sub>R<sub>2</sub>, m. 134°; 1,2,4-MeOC<sub>6</sub>H<sub>3</sub>R<sub>2</sub>, m. 75°;  
1,2,5-isomer, m. 89°; 1,4,2,5-Me(MeO)C<sub>6</sub>H<sub>2</sub>R<sub>2</sub>, m. 106-7°;  
1,2,4-EtOC<sub>6</sub>H<sub>3</sub>R<sub>2</sub>, b16 162-4°, m. 56°; 1,3,4,6-(MeO)<sub>2</sub>C<sub>6</sub>H<sub>2</sub>R<sub>2</sub>,  
m. 125° [bis-(methylurethane), m. 154°]; 1,4,2,  
5-isomer, m. 180-1°; 1,2,4-PrOC<sub>6</sub>H<sub>3</sub>R<sub>2</sub>, b15 164-5°; 1-iso-BuO  
homolog, b14 180-1°; 1,4,2,5-(EtO)C<sub>6</sub>H<sub>2</sub>R<sub>2</sub>, m. 128°;  
(p-RC<sub>6</sub>H<sub>4</sub>N<sub>2</sub>)<sub>2</sub>, m. 158-61° [bis(methylurethane), m. 241-3°];  
p-[4, 2-RMeC<sub>6</sub>H<sub>3</sub>N:N]C<sub>6</sub>H<sub>4</sub>R, m. 123-5° [bis(methylurethane), m.  
244-8°]; 1, 4-(p-RC<sub>6</sub>H<sub>4</sub>N:N)C<sub>6</sub>H<sub>10</sub>R, m. 177-8°,  
[bis(methylurethane), m. 210-12°]; p-(o-RC<sub>6</sub>H<sub>4</sub>O)C<sub>6</sub>H<sub>4</sub>R, m.  
60-2°; (p-RC<sub>6</sub>H<sub>4</sub>)<sub>2</sub>O, b5 196°, m. 66-8°;

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(*o*-RC<sub>6</sub>H<sub>4</sub>OCH<sub>2</sub>)<sub>2</sub> and (*o*-RC<sub>6</sub>H<sub>4</sub>CH<sub>2</sub>CH<sub>2</sub>)<sub>2</sub>O (prepared but not characterized). The following diisocyanato derivs. of naphthalene were prepared: 1, 4, m. 67-70° [bis(methylurethane), m. 220-2°]; 1, 5, m. 130-2° (subliming in high vacuum) [bis(methylurethane), m. 245-6°]; 2, 6, m. 152-4° [bis(methylurethane), m. 244-6°]; 2, 7, m. 152-3°; (2, 1-RC<sub>10</sub>H<sub>6</sub>)<sub>2</sub>, m. 132-3° [bis(methylurethane), m. 185-7°]. The following diisocyanato derivs. of Ph<sub>2</sub> were formed: 2, 4', b0.5 140-50° [bis(methylurethane), m. 171-2°]; 4, 4' (X), b0.5 150-60° [bis(methylurethane), m. 244°]; 3, 3'-di-Me derivative of X, m. 68-9° [bis(methylurethane), m. 208°]; 3, 3'-di-MeO derivative of X, m. 121-2° [bis(methylurethane), m. 215-16°]; 2-NO<sub>2</sub> derivative of X, m. 119-21° [bis(methylurethane), m. 211-12°]. The following diisocyanates were formed: (p-RC<sub>6</sub>H<sub>4</sub>)<sub>2</sub>CH<sub>2</sub>, b0.2 170°, m. 46° (also given as 37.6°) [bis(dimethylurethane), m. 183-3.5°]; (2, 4-MeRC<sub>6</sub>H<sub>3</sub>)<sub>2</sub>CH<sub>2</sub>, b5 208-11°, m. 65-7°; p-R(C<sub>6</sub>H<sub>4</sub>2CMe<sub>2</sub>, b5.6 212°, m. 91.5°; (2,5,4-Me<sub>2</sub>RC<sub>6</sub>H<sub>2</sub>)<sub>2</sub>CH<sub>2</sub>, m. 127.6°; (p-RC<sub>6</sub>H<sub>4</sub>)<sub>2</sub>CH<sub>6</sub>11(?), b0.1 195-200° [bis(methylurethane), m. 173°]; [4, 3-R(MeO) C<sub>6</sub>H<sub>3</sub>]<sub>2</sub> CH<sub>2</sub>, m. 97-8°; [3, 4-R(MeO) C<sub>6</sub>H<sub>3</sub>]<sub>2</sub> CH<sub>2</sub>, m. 90-3°; [3, 4-R(EtO)C<sub>6</sub>H<sub>3</sub>]<sub>2</sub> CH<sub>2</sub>, m. 74°; [4, 2, 5-RMe (MeO) C<sub>6</sub>H<sub>2</sub>]<sub>2</sub>CH<sub>2</sub>, m. 128-9°; [4, 3-RC<sub>1</sub>C<sub>6</sub>H<sub>3</sub>]<sub>2</sub> CMe<sub>2</sub>, m. 100-1°; (m-RC<sub>6</sub>H<sub>4</sub>)<sub>2</sub> CO, m. 118-20°; 1,2,4-PhCH<sub>2</sub>CH<sub>2</sub>C<sub>6</sub>H<sub>3</sub>R<sub>2</sub>, b20 223-7° [bis(methylurethane), m. 154-5°]; m-(p-RC<sub>6</sub>H<sub>4</sub>)<sub>2</sub> CHC<sub>6</sub>H<sub>4</sub>NO<sub>2</sub> (not characterized) [mono- or bis(phenylurea), sinters about 145°]; p-isomer (not characterized) [mono- or bis(phenylurea) (?), m. 224-6°]; (4, 2, 5-RMe<sub>2</sub>C<sub>6</sub>H<sub>2</sub>)<sub>2</sub> CHPh, m. 116-18°. The following diisocyanato derivs. of fused rings and of aromatic S compds. were formed: 2, 7-R<sub>2</sub>-fluorene, m. 133°; 2, 6-R<sub>2</sub>-anthraquinone, m. 250-2°; 9-ethyl-2, 7-R<sub>2</sub>-carbazole (S. gives 3, 6), m. 122-3°; 3, 8-R<sub>2</sub>-pyrene, m. 234° [bis-(methylurethane), m. 264°]; 5, 11-R<sub>2</sub>-chrysene, m. 274° (S. gives 2, 8). The following diisocyanates were prepared from aromatic S-containing compds. 1,2,4-PhSC<sub>6</sub>H<sub>3</sub>R<sub>2</sub>, m. 73-4°; (p-RC<sub>6</sub>H<sub>4</sub>S)<sub>2</sub>, m. 58-60°; (p-RC<sub>6</sub>H<sub>4</sub>)<sub>2</sub>SO<sub>2</sub>, m. 154° [bis-(methylurethane), m. 262-4°]; XI, m. 166-8° [bis(methylurethane), m. 235-7°]; 1,2,4-(p-MeC<sub>6</sub>H<sub>4</sub>SO<sub>2</sub>) C<sub>6</sub>H<sub>3</sub>R<sub>2</sub> (impure); p-(3, 4-RMeC<sub>6</sub>H<sub>3</sub>SO<sub>3</sub>) C<sub>6</sub>H<sub>4</sub>R, m. 67.5°; 4-MeO analog, m. 104-6°; (5, 2-RMeC<sub>6</sub>H<sub>3</sub>S)<sub>2</sub>, m. 77-8°; 2, 5-isomer, m. 74-6°; 3, 4-isomer, m. 88°; [3, 4-R(MeO)-C<sub>6</sub>H<sub>3</sub>S]<sub>2</sub>, m. 66-7°; 4, 3-isomer, m. 101° [3, 4-R(MeO)C<sub>6</sub>H<sub>3</sub>]<sub>2</sub> SO<sub>2</sub>, m. 165°; 1, 3, 4-(3, 4-RMeC<sub>6</sub>H<sub>3</sub>SO<sub>2</sub> NH)C<sub>6</sub>H<sub>3</sub>MeR, m. 191-6°; (p-RC<sub>6</sub>H<sub>4</sub>SO<sub>2</sub>NHCH<sub>2</sub>)<sub>2</sub> (m. poorly 178°); [4, 3-R(MeO)C<sub>6</sub>H<sub>3</sub>CH<sub>2</sub>]<sub>2</sub>S (not characterized); [3, 4-R(MeO)C<sub>6</sub>H<sub>3</sub>SCH<sub>2</sub>]<sub>2</sub>, m. 118°; [4, 3-R(MeO)C<sub>6</sub>H<sub>3</sub>CH<sub>2</sub>]<sub>2</sub> SO<sub>2</sub> (not characterized); [3, 4-(MeO)C<sub>6</sub>H<sub>3</sub>SO<sub>2</sub>CH<sub>2</sub>]<sub>2</sub>, m. 265-7°; [3, 4-(MeO)C<sub>6</sub>H<sub>3</sub>SO<sub>2</sub>NHCH<sub>2</sub>]<sub>2</sub>, m. 192°. The following triisocyanates were formed: 1, 2-4, 6-MeC<sub>6</sub>H<sub>2</sub>R<sub>3</sub>, b1-3 133-9°, m. 75° [tris(methylurethane), m. 195°]; 1, 3, 5, 2, 4, 6-Me<sub>3</sub>C<sub>6</sub>R<sub>3</sub>, m. 93° [tris(methylurethane), m. 284°]; 1, 3, 7-C<sub>10</sub>H<sub>5</sub>R<sub>3</sub>, m. 162-3° [tris(methylurethane), m. 217°]; p-(2, 4-R<sub>2</sub>C<sub>6</sub>H<sub>3</sub>) C<sub>6</sub>H<sub>4</sub>R, b1.5-2 194-8°, m. 71°; p-(2, 4-R<sub>2</sub>C<sub>6</sub>H<sub>3</sub>CH<sub>2</sub>) C<sub>6</sub>H<sub>4</sub>R, undistillable; p-(2, 4, 3-R<sub>2</sub>MeC<sub>6</sub>H<sub>2</sub>CH<sub>2</sub>) C<sub>6</sub>H<sub>4</sub>R, b4 224°, m. 75-7° [tris(methylurethane), m. 167°]; (p-RC<sub>6</sub>H<sub>4</sub>)<sub>3</sub>CH, m. 89-90°. The following were also formed: (4, 2, 5-Me<sub>2</sub>RC<sub>6</sub>H<sub>2</sub>)<sub>2</sub> CH<sub>2</sub>, m. 154-6°; (p-RC<sub>6</sub>H<sub>4</sub>)<sub>2</sub>NCOCl, b2-3 230-4°, m. 74-77° [bis(methylurethane), m. 182-4°]. The following dimers of the type formula [1,2,4-RC<sub>6</sub>H<sub>3</sub>(NCO)<sub>2</sub>]<sub>2</sub> were prepared: R = Cl, m. 177°; Me, m. 156°; Et, m. 148°; EtO, m. 185-6°; Me<sub>2</sub>CH, m. 125°; Me<sub>2</sub> CHO, m. 158°; iso-BuO, m. 176°. Also formed was [1,2,5-MeC<sub>6</sub>H<sub>3</sub>(NCO)<sub>2</sub>]<sub>2</sub>, m. 186°.

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| COST IN U.S. DOLLARS                       | SINCE FILE ENTRY | TOTAL SESSION |
|--|------------------|---------------|
| FULL ESTIMATED COST                        | 99.06            | 99.27         |
| DISCOUNT AMOUNTS (FOR QUALIFYING ACCOUNTS) | SINCE FILE ENTRY | TOTAL SESSION |
| CA SUBSCRIBER PRICE                        | -21.48           | -21.48        |

FILE 'USPATFULL' ENTERED AT 13:55:18 ON 21 JUN 2004  
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CA INDEXING IS CURRENT THROUGH 17 Jun 2004 (20040617/UPCA)

ISSUE CLASS FIELDS (/INCL) CURRENT THROUGH: 17 Jun 2004 (20040617/PD)

REVISED CLASS FIELDS (/NCL) LAST RELOADED: Apr 2004

USPTO MANUAL OF CLASSIFICATIONS THESAURUS ISSUE DATE: Apr 2004

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=> s phosgenation  
L16 3991 PHOSGENATION

=> s phosgene  
L17 19237 PHOSGENE

=> s l16 and l17  
L18 1912 L16 AND L17

=> s l18 and diamine  
68324 DIAMINE  
L19 691 L18 AND DIAMINE

=> s l19 and isocyanate  
59036 ISOCYANATE  
L20 602 L19 AND ISOCYANATE

=> s l20 and spray  
213596 SPRAY

10670409-isocyanate

L21 109 L20 AND SPRAY

=> s l21 and nozzles  
107350 NOZZLES

L22 2 L21 AND NOZZLES

=> s "spray nozzles"  
213596 "SPRAY"  
107350 "NOZZLES"  
L23 13823 "SPRAY NOZZLES"  
("SPRAY" (W) "NOZZLES")

=> s l23 and l20

L24 0 L23 AND L20

=> d 1-2 bib abs l22

L22 ANSWER 1 OF 2 USPATFULL on STN

AN 2003:282455 USPATFULL

TI Stabilized aqueous crosslinker dispersions

IN Rische, Thorsten, Unna, GERMANY, FEDERAL REPUBLIC OF  
Naujoks, Karin, Odenthal, GERMANY, FEDERAL REPUBLIC OF  
Meixner, Juergen, Krefeld, GERMANY, FEDERAL REPUBLIC OF  
Feller, Thomas, Solingen, GERMANY, FEDERAL REPUBLIC OF  
Konig, Eberhard, Leverkusen, GERMANY, FEDERAL REPUBLIC OF

PI US 2003198796 A1 20031023

AI US 2003-341977 A1 20030114 (10)

PRAI DE 2002-10201545 20020117

DT Utility

FS APPLICATION

LREP BAYER POLYMERS LLC, 100 BAYER ROAD, PITTSBURGH, PA, 15205

CLMN Number of Claims: 20

ECL Exemplary Claim: 1

DRWN No Drawings

LN.CNT 784

CAS INDEXING IS AVAILABLE FOR THIS PATENT.

AB The present invention relates to a water-dispersible crosslinker composition containing

A) at least one hydrophilically-modified, blocked polyisocyanate,

B) at least one stabilizing agent containing

a) at least one amine containing a structural unit corresponding to formula (I) ##STR1##

which does not contain hydrazide groups,

b) at least one compound containing a structural unit corresponding to formula (II)

--CO--NH--NH-- (II)

and

c) optionally a stabilizing component other than a) and b), and

C) optionally an organic solvent.

The present invention also relates to an aqueous solution or dispersion containing this crosslinker composition, to aqueous coating compositions containing this crosslinker composition and to glass fibers coated with

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this coating composition.

CAS INDEXING IS AVAILABLE FOR THIS PATENT.

L22 ANSWER 2 OF 2 USPATFULL on STN  
AN 76:40550 USPATFULL  
TI Colored poly (urethane) urea powders  
IN Weber, Karl-Arnold, Leverkusen, Germany, Federal Republic of  
Reiff, Helmut, New Martinsville, WV, United States  
Dieterich, Dieter, Leverkusen, Germany, Federal Republic of  
PA Bayer Aktiengesellschaft, Leverkusen, Germany, Federal Republic of  
(non-U.S. corporation)  
PI US 3970601 19760720  
AI US 1975-573491 19750501 (5)  
PRAI DE 1974-2425810 19740528  
DT Utility  
FS Granted  
EXNAM Primary Examiner: Welsh, M. J.  
LREP Pope, Lawrence S., Harsh, Gene  
CLMN Number of Claims: 5  
ECL Exemplary Claim: 1  
DRWN No Drawings  
LN.CNT 1198  
CAS INDEXING IS AVAILABLE FOR THIS PATENT.  
AB The instant invention relates to thermoplastic polyurea powders,  
optionally containing urethane groups, characterized by  
A. a smooth, substantially spherical or lenticular surface,  
B. an average particle diameter of 5 to 1,000 $\mu$ , preferably 10 to  
200 $\mu$ ,  
C. a urea group content, and, optionally, a urethane group content, of 8  
to 30% by weight,  
D. an ionic group content of 0.01-0.15 milliequivalents per gram of  
solids, preferably 0.02-0.1 milliequivalent per gram and  
E. 0.1 to 10% by weight, preferably 0.2 to 5% by weight, of an organic  
dye incorporated in the polymer molecule by way of at least one urethane  
and/or urea group,

And the process of manufacture thereof.

CAS INDEXING IS AVAILABLE FOR THIS PATENT.